# METALLURGIA

THE BRITISH JOURNAL OF METALS

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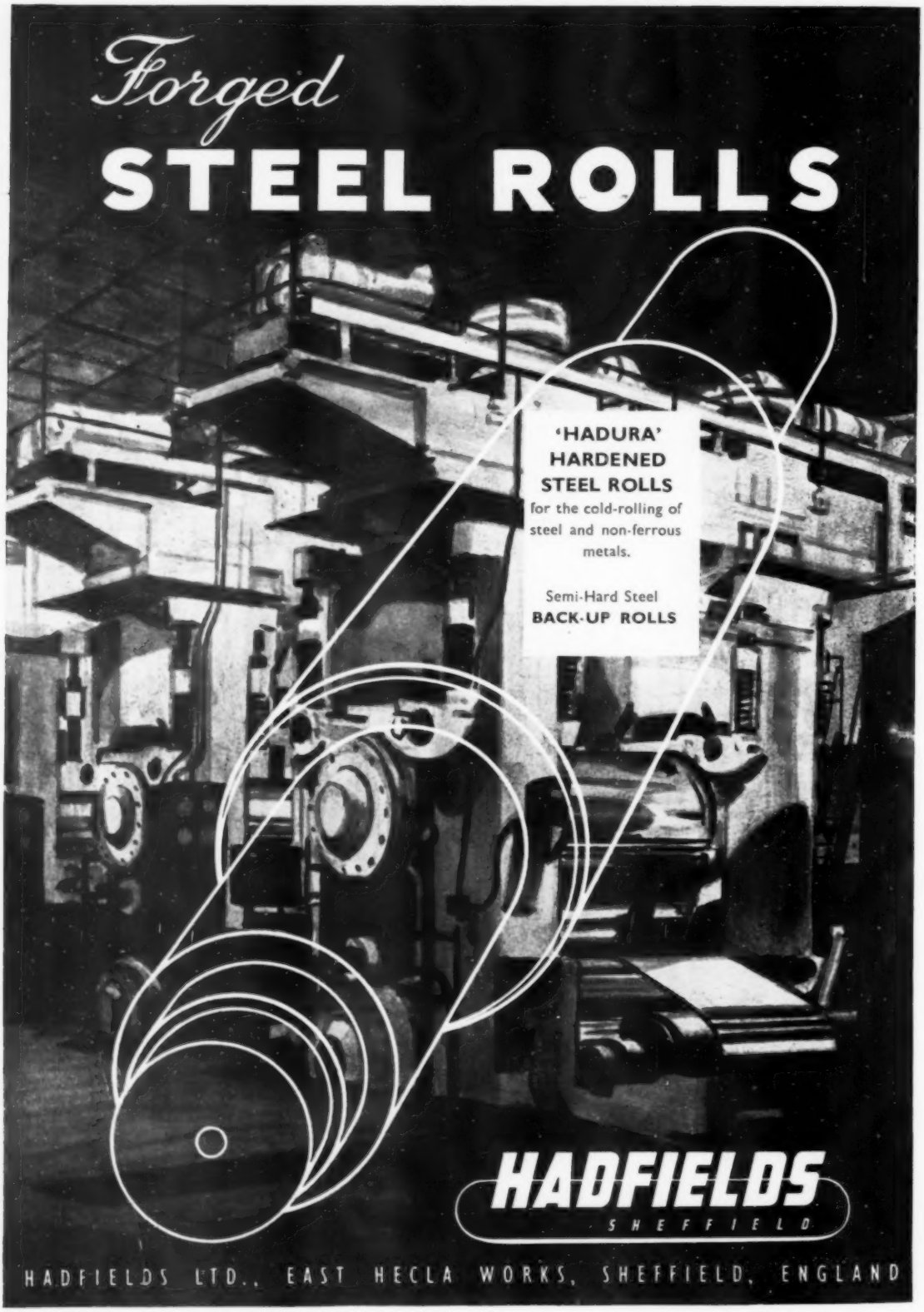
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# METALLURGIA

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INCORPORATING THE METALLURGICAL ENGINEER


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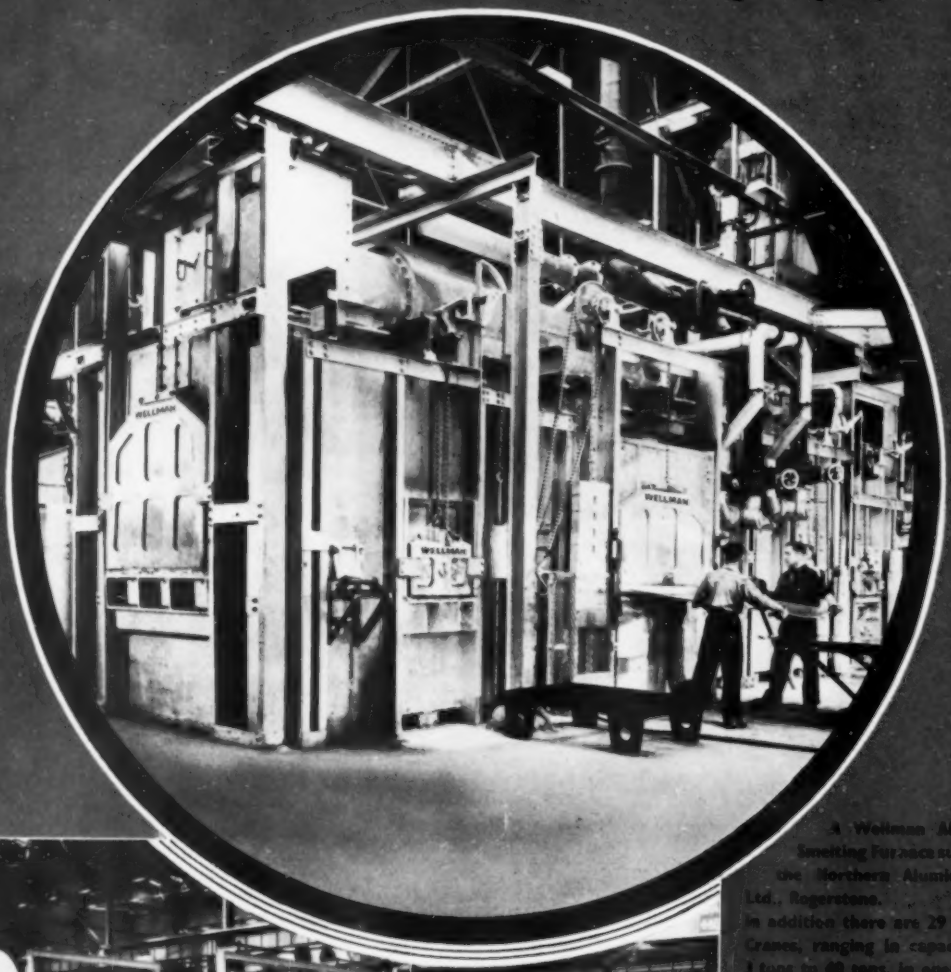
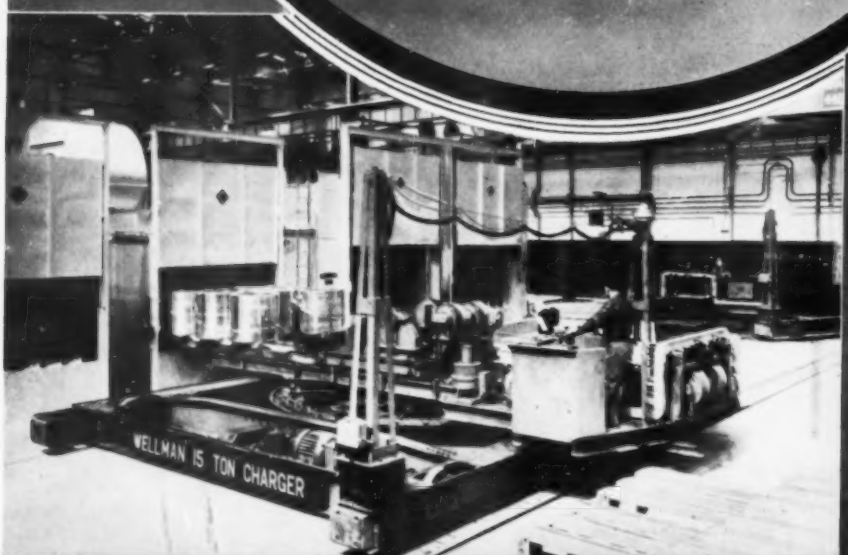
# WELLMAN

## Furnaces and Mechanical Handling Equipment



A Wellman Aluminium Smelting Furnace supplied to the Northern Aluminium Co. Ltd., Rogerstone.

In addition there are 29 Wellman Cranes, ranging in capacity from 1 tons to 40 tons, in operation at these Works.




Wellman 15 ton Finger Type Chargers and Turntable in service at the British Aluminium Co. Ltd., Falkirk. This installation also includes a 10,000 lbs. Wellman Slab Discharger and a 18,000 lbs. Block Handling Crane.

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# METALLURGIA

THE BRITISH JOURNAL OF METALS

INCORPORATING THE "METALLURGICAL ENGINEER"

MAY, 1955 Vol. LI. No. 307


## Handling in the Small Firm

EVERY time material is handled in the factory something is added to its cost, and nothing to its value: this is true whether the handling is by machinery or manpower—a point not always realised. There is here, then, a standing challenge to every industrial organisation to cut this cost, and to improve productivity, by paying greater attention to matters of handling.

Out of a total wage bill for British manufacturing industries of about £2,500 million a year, between one-sixth and one-third can be attributed to handling. Accidents causing loss of time cost industry in one year alone £70 million: 28% of such accidents—£19 million worth—were due to handling. Elimination of unnecessary handling, so that materials could flow uninterruptedly to and from the worker, could raise productivity by 15% without any change in the existing production machinery. These facts appear in "The Handling of Materials," which has been published recently (price 9d.) by The British Productivity Council, 21, Tothill Street, London, S.W.1, as No. 3 in its Action Pamphlet series. They provide some of the leading reasons for a systematic examination of all handling in any enterprise.

There is a tendency to think of materials handling in terms of mass production factories, with large fleets of fork-lift trucks, extensive mechanised conveyors, and automatic processing. There are such factories, of course, but it should be realised that excessive or haphazard handling causes unnecessary expenditure in any firm, however small, and whatever its production. Moreover, small firms represent an appreciable proportion of the nation's production capacity—75% of the manufacturing establishments in the country have less than 100 employees, and 96% have less than 500—so that reduction in costs and increases in output in this section of industry are of considerable national significance.

Ever since goods have been made and distributed, the work of lifting and moving them has been done by men, and in many instances the size of unit, such as the sack of flour or the pig of iron, has been determined by man's lifting capacity. Much of this kind of work is to-day a most wasteful use of manpower, as it inevitably results in unnecessary fatigue and, in some instances, takes up the time of skilled men, who could be more profitably employed on other tasks. As for men, so for materials: the less handling there is, and the better the methods used, the less likely are the goods to suffer damage.

In practice, mechanisation accounts for only a proportion of the time saved by good materials handling—a fact which should prove a source of encouragement to small firms inclined to regard such refinements as beyond their financial resources. Considerable savings come from the elimination of various forms of wasted effort, many of which are not recognised until a firm begins to give serious attention to its handling problems. In the same way, studies made with the object of improving handling methods often indicate changes that might beneficially be made in plant layout. Furthermore, once a management has set itself the aim of making its handling efficient—and, as a preliminary step, has had to find out exactly what is happening at every stage of operations in the factory—it is on the way to being able to assess and control its costs more accurately.

Factory space made available for more production is one of the greater benefits of good materials handling. Many firms housed in old buildings, not designed for the present purpose, can do much to overcome their limitations by the use of modern handling methods. In fact, planned handling can save so much space that firms may even be able to avoid the cost of extra buildings.

Lower costs are implicit in most of the advantages set out above, but there is too a direct saving in costs, as is clear from the axiom that handling adds nothing but cost to the finished product. Handling costs can rise as high as 85% of the cost of production, and it is not unusual for 50 tons of material to be handled for every ton of finished product. The expenditure incurred in installing a good handling system may be negligible, but whatever it is, it should be regarded as an investment.

Every materials handling problem is an individual one, and the smaller firm, which has not previously paid special attention to materials handling, may be in need of guidance as to how to set about studying the problem. A number of suggestions found helpful by firms which have already tackled handling problems efficiently are referred to in the pamphlet.

Before drastic alterations are made, or capital expenditure is considered, a complete and possibly lengthy, study of the problem should be made by a selected member of the production staff with a good knowledge of the company and its products, and an ability to get on with people. Similarly, before any action is taken which affects the workers, the management should make clear to them what it is trying to do and why.

Attention should be focused on points where expenditure is greatest, and where substantial savings may therefore be possible. A preliminary survey of the movement of materials through the factory may be only rough, but if the cost in time and labour is estimated from it, the amount will almost certainly be higher than expected. Furthermore, the survey will probably indicate one or two points at which the waste is particularly great. With a subsequent and more detailed survey before it, the management can plan the improvements it is proposed to make, such as alterations to layout and installation of handling equipment.

On the matter of equipment, it is, perhaps, not so well known as it should be that every device and all the knowledge and skill available to industry in the United States is available also to British industry. There is, therefore, no reason why British firms should fall behind their American counterparts in taking full advantage of modern handling methods.

## Co-operative Research in Ferrous Powder Metallurgy

DECISIONS of importance to all those interested in the development of ferrous powder metallurgy were reached at a recent London meeting, convened by the Metallurgy (General) Division of the British Iron and Steel Research Association. Those taking part included representatives from industry, together with research and academic workers in the metallurgical field.

The Chairman (Dr. M. L. Becker, Head of B.I.S.R.A.'s Metallurgy Division) opened the meeting by extending a welcome to those present, who had been invited to attend in order to discuss problems in the field of ferrous powder metallurgy, and to consider the desirability of co-operation and mutual assistance for the extension of knowledge in this field. Dr. Becker emphasised that the meeting was purely informal, and had been called by B.I.S.R.A. chiefly because suggestions had been put forward urging the Association to conduct investigations on the subject, a proposal which it seemed appropriate to consider in relation to the Powder Metallurgy Symposium recently held jointly by the Iron and Steel Institute and the Institute of Metals.

Some concern was expressed at the limitations imposed by economic considerations on the expansion of the industry, and it was generally agreed that there were grounds for co-operative research into the production of commercially acceptable powder. The need for a wider exchange of information about the behaviour of different types of ferrous metal powders was discussed, but it was decided that the value of fundamental research needed further demonstration to justify broad industrial support.

Although research into the processing of ferrous powders is already carried out by certain sections of the industry—and to a limited extent by the universities—it was felt that certain of the more important questions awaiting solution were economically unattractive to individual firms. The techniques of pressing and sintering, including hot pressing, as well as the problem of die wear, were examples of this kind, and the meeting concluded that further data on certain aspects of processing was undoubtedly desirable.

There was a prolonged discussion on the question of establishing a co-operative research organisation, which might cover ferrous, non-ferrous and ceramic powder interests, and eventually the proposition was adopted that "... the organisations represented view with favour the suggestion from B.I.S.R.A. that an Association should be set up for co-operative research in the powder metallurgy industry."

The Chairman explained that B.I.S.R.A. would be unlikely to be able to conduct any preliminary investigation, as had been done in the analogous case of the Cutlery Research Organisation, unless interested parties made funds available. The possibility was mentioned, however, of support from the D.S.I.R., especially in view of the report of the Commission which recently visited America to study powder metallurgy practice.

The members of this Commission are being invited by the Ministry of Supply to address a gathering of British industrialists on their tour and the recommendations arising therefrom. It is expected that this function will be held in the late spring, and the meeting felt that it would afford an excellent opportunity for the tentative establishment of a co-operative research body which might well serve the whole powder metallurgy industry.

The conclusions reached by the meeting will undoubtedly be echoed by many other firms and organisations engaged in the same field. Powder metallurgy is an industrial technique with great potentialities, and there would certainly appear to be an overwhelming case for supporting its economic development with a properly organised research programme. Industry will no doubt watch progress in the matter with considerable interest.

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## Discussions on the Physics of Non-Destructive Testing

THE Non-Destructive Testing Group of the Institute of Physics has arranged for discussions to take place, at various times during the coming year, on the physics of non-destructive testing. The initial discussions will be concerned with the broad fields covered by the three following subject headings: (1) the physics of testing metals; (2) the physics of testing non-metals; and (3) the physics of medical testing. It is proposed that the discussions should be followed subsequently by lectures and papers dealing with detailed investigations which come within the scope of these three subjects.

In the case of the testing of metals and non-metals, an effort is being made to stimulate active interest in the study of the relationship between the physical properties of materials and their constitution and structure. It is envisaged that the relationships so established will serve as a basis for the development of new testing techniques. In addition, it is hoped that the information gained by the establishment of these relationships will be of use to those physicists who are engaged on fundamental work concerned with the understanding of physical properties.

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## Mr. J. W. Howlett Honoured

ON Tuesday, March 29th, Mr. J. W. Howlett, Chairman and Managing Director of the engineering firm of Wellworthy, Ltd., Lymington, Hants., was made an Honorary Freeman of the ancient borough of Lymington, in recognition of "his eminent services to the borough and his outstanding contribution to its welfare and development over a period of many years."

Mr. Howlett, who is 72 years old, started Wellworthy in 1918 with seven employees, of which he was one. To-day, there are two large and still expanding factories at Lymington, a modern foundry at Ringwood, and another factory at Salisbury. The firm started by making piston rings, and then went on to make pistons, cylinder liners, aero engine castings, and other components. It is the sole licensee in this country for the Al-Fin process for bonding steel to aluminium.

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## N.P.L. Open Day

THE National Physical Laboratory, Teddington, is holding an Open Day on Friday, May 20th. Admission is by ticket only and a limited number of invitations for the afternoon period, 1-30–5-30 p.m., are still available on application to the Director.

# Nickel Alloys Made by Powder Metallurgy Techniques

*The closer control over composition possible in the production of nickel and nickel alloys by powder metallurgy methods results in an improvement in properties which is particularly valuable in the electronic field. Particulars are given of commercially available materials*

POWDER metallurgy is now a well established technique for the production of metals and alloys for specialised uses, and its application to wrought nickel and nickel alloys has been found to offer a number of advantages, both in the manufacturing processes and in the properties of the final products. Henry Wiggin & Co., Ltd., have recently installed equipment in their Birmingham works for the manufacture of nickel and nickel alloy strip by this technique, to meet the requirements of the many industries in which materials of closely controlled composition and properties are required.

## Manufacturing Procedure

The basic raw materials are carbonyl nickel and iron powders, which are essentially of high purity. Other materials used include cobalt and molybdenum powders prepared by hydrogen-reduction of pure oxides and O.H.F.C. copper powder produced by atomisation. Table I indicates the degree of purity available in the raw materials. In view of the nature of the production process, the level of purity in the final alloy can be expected to be at least as high as in the original constituents: it may even be improved owing to removal of oxygen and carbon during processing. This is one of the important advantages of the powder-metallurgy method over conventional melting processes, in which contamination is invariably introduced from crucible linings, deoxidants, etc.

In weighing out the constituent powders for a mix it is possible in practice to maintain an accuracy of at least 0·05%, and the composition can, therefore, be maintained within very close limits, as there are no losses such as normally occur in melting. Thorough mixing of the component powders is carried out by tumbling for several hours to obtain homogeneity. This treatment has been found completely satisfactory, even when several constituents are involved, some of which may be present in only minor proportions. The mixed powders are then pressed into compacts at a pressure of between 20 and 35 tons/per sq. in., according to the composition. The weight of these compacts is about 15 lb.

The compacts are sintered in an electrically-heated tube furnace, in an atmosphere of hydrogen. Special precautions are taken in the purification and drying of the furnace atmosphere to ensure freedom from contamination. An initial heat treatment is carried out at a temperature of 600° C. for several hours, in order to remove the bulk of impurities such as carbon, oxygen, and nitrogen in the porous compact. This is followed by sintering at a temperature of about 1200° C. for several hours.

After sintering the compacts are forged into either flats or bars, which can subsequently be processed into strip, rod, or wire, by normal hot- and cold-working operations. Special precautions are taken to avoid contamination throughout the processing operations. For rolling to strip the sintered compacts are forged into flats about ¼ in. thick, which are first hot-rolled and finally cold-rolled to size. For the production of rod and wire the sintered compacts are forged to rectangular bars, hot-rolled to rod, and drawn down to wire.

TABLE I.—ANALYSES OF RAW MATERIALS.

| Powder | Type | Average Particle Size (micron) | Impurities | | | | | |
|---|---|---|---|---|---|---|---|---|
| | | | O% | C% | S% | Ni% | Fe% | Others % |
| Nickel | Carbonyl grade A | 4·5 | 0·065 | 0·065 | 0·002 | — | 0·02 | Si and Mg 0·005* |
| Iron | Carbonyl grade MCP | 7·0 | 0·122 | 0·009 | 0·002 | 0·35 | — | Si and Mg 0·005* |
| Cobalt | Hydrogen reduced | <0·5 | 0·6 | 0·04 | 0·080 | 0·09 | 0·09 | Pb <0·005 Si 0·01–0·02 |
| Molybdenum | Hydrogen reduced | 3 | 0·12 | n.d. | n.d. | n.d. | n.d. | Si 0·002 Mg 0·005* |
| Copper | Atomised O.F.H.C. | 17 | 0·25 | n.d. | 0·006 | trace | 0·037 | Si 0·002 Pb 0·010 Sn 0·025 Zn 0·024 |
| Tungsten | Hydrogen reduced | <0·5 | 0·1 | n.d. | trace | n.d. | n.d. | Si 0·02–0·05 |

* Spectrographic data.
n.d. = not determined.

After processing, the density of products produced by powder metallurgy compares very favourably with those made by normal methods of melting. The final product has a much lower content of non-metallic inclusions and other impurities than is characteristic of alloys prepared by normal melting techniques.

## Advantages of the New Materials

The production of nickel and nickel alloys by powder metallurgy techniques offers a number of advantages. The composition can be controlled more accurately than is possible in normal melting methods. Thus it is possible to exercise much closer control over the properties and, by virtue of the much higher purity, some quite remarkable improvements can be obtained.

These advantages are particularly useful in the electronics industry, where there is a need for alloys with physical properties conforming closely to stringent specifications. Such uses include metals for thermionic valve construction, alloys with special expansion characteristics and magnetic materials.

Some notes are given below on the alloys already being produced using powder metallurgy techniques, and an indication is given of those applications where they are already proving useful.

## High-Purity Nickel

From the description of the process, it will be appreciated that in the manufacture of H.P.M. (high purity)

TABLE II.—COMPARATIVE SPECIFICATIONS FOR VARIOUS GRADES OF WROUGHT NICKEL.

| Grade | C% | Si% | Cu% | Fe% | Mn% | Mg% | Al% | S% | Ni + Co% |
|---|---|---|---|---|---|---|---|---|---|
| H.P.M. Nickel | 0·02 max. | 0·01 max. | 0·01 max. | 0·04 max. | 0·01 max. | 0·005 max. | 0·005 max. | 0·005 max. | 99·9 min. |
| H.P.A. Nickel | 0·10 max. | 0·03 max. | 0·04 max. | 0·05 max. | 0·02 max. | 0·01 max. | 0·01–0·05 | 0·005 max. | 99·5 min. |
| H.P.B. Nickel | 0·10 max. | 0·15–0·25 | 0·04 max. | 0·05–0·10 | 0·10 max. | 0·01 max. | 0·02 max. | 0·005 max. | 99·5 min. |
| "O" Nickel | 0·04 max. | 0·02–0·05 | 0·10 max. | 0·20 max. | 0·15 max. | 0·05–0·10 | — | 0·005 max. | 99·5 min. |
| G.F.A. Nickel | 0·05 max. | 0·03–0·07 | 0·20 max. | 0·25 max. | 0·05 max. | 0·07–0·15 | — | 0·005 max. | 99·0 min. |

Nickel it is unnecessary to make any specific additions of reducing agents such as magnesium, silicon or manganese which are normally added during melting as deoxidisers. The purity thus obtained is demonstrated by the specifications given in Table II.

On account of the higher purity, the temperature coefficient of electrical resistivity is considerably higher, and the specific resistance lower, than in the case of melted alloys. Results are shown in Table III., these were obtained on 0·020 in. diameter wire drawn from four independently produced compacts of nickel. The tests were carried out on samples which had been strand bright-annealed, stabilised by heating at 450° C., followed by slow cooling.

Where no provision is made for the replenishment of hydrogen in hydrogen thyratrons, it is essential to avoid reduction in the gas pressure due to combination with impurities in the components of the valve. In such cases, H.P.M. Nickel has proved very satisfactory for anodes, grids and support structures.

Nickel and nickel alloys produced by powder metallurgy may be suitable for the sleeves of indirectly heated oxide-coated cathodes where very high purity is essential, such as for valves where a satisfactory level of emission must be maintained over long periods of operation. These conditions apply, for instance, in submarine repeater valves or in valves which are run for long periods at cut-off conditions, yet must deliver a good emission current when required. It is probable that the presence of silicon, in the quantities that usually occur in melted alloys, has a deleterious effect on emission under certain circumstances, and production by powder metallurgy is, therefore, almost essential where extremely low silicon contents are required.

To establish a steady emission level, cathodes made from such high-purity nickel may require a longer activating schedule than those made from "O" Nickel, which contains sufficient reducing agents in the form of magnesium and silicon to promote rapid cathode activity. It is not easy to introduce into powder metallurgy alloys activating agents of this type, but recent experimental work indicates that tungsten shows some promise as an activating agent. Silicon-free tungsten alloys can be readily produced by powder metallurgy methods, and compositions containing 2% and 3·5% tungsten are currently in production. H.P.M. Nickel is also useful in other types of cathode of composite construction, in which an activating medium is disposed separately from the main structure.

TABLE III.—ELECTRICAL RESISTIVITY OF H.P.M. NICKEL.

| Sample No. | Temperature Coefficient of Resistance (0—100° C.) | Specific Resistance at 20° C. (microhm-cm.) |
|---|---|---|
| 1 | 0·00685 | 7·178 |
| 2 | 0·00682 | 7·171 |
| 3 | 0·00684 | 7·202 |
| 4 | 0·00685 | 7·160 |
| Average for Other Grades | 0·005 | 9·0 |

## Controlled Expansion Alloys

Although the characteristics of the nickel-iron alloys are primarily determined by the nickel : iron ratio, relatively small amounts of such elements as chromium, manganese, silicon and copper also have a marked effect on both expansion properties and the inflection temperature. With the powder metallurgy technique, the two-fold advantage of control of composition and absence of impurities makes it possible to produce alloys to meet specific requirements of expansion, and to much closer tolerances than heretofore.

Table IV shows the mean coefficient of a range of nickel-iron alloys with nickel contents between 36 and 51%. It will be noted that powder-metallurgy nickel-iron alloys tend to have lower expansion values and a higher inflection temperature than corresponding commercial alloys.

TABLE IV.—MEAN COEFFICENT OF EXPANSION FOR NICKEL-IRON ALLOYS PRODUCED BY POWDER METALLURGY (cm./cm./° C. × $10^{-6}$).

| Temperature Range | Nickel Content | | | | | |
|---|---|---|---|---|---|---|
| | 36% | 39% | 41% | 44% | 48% | 51% |
| 20–100° C. | 0·9 | 2·4 | 3·5 | 6·6 | 8·2 | 9·3 |
| 20–200° C. | 1·9 | 2·3 | 3·6 | 6·4 | 8·5 | 9·6 |
| 20–300° C. | 4·5 | 3·0 | 3·9 | 6·3 | 8·5 | 10·0 |
| 20–400° C. | 7·3 | 5·2 | 5·3 | 6·3 | 8·5 | 10·0 |
| 20–500° C. | 9·2 | 7·3 | 7·2 | 7·6 | 8·7 | 9·9 |
| 20–600° C. | 10·5 | 8·8 | 8·7 | 8·9 | 9·7 | 10·4 |

The powder metallurgy process is equally applicable to the production of ternary alloys, such as nickel-cobalt-iron of the Nilo K type for sealing into medium-hard borosilicate glasses.

## Magnetic Alloys

A considerable amount of research has been devoted to the production of high-permeability magnetic alloy strip by powder metallurgy. It has been shown in a recent paper by Walker, Worn & Walters* that marked improvement can thus be obtained in the magnetic properties of an alloy of the composition Ni 77%, Fe 14%, Mo 4%, Cu, 5%.

The permeability of these alloys when subjected to a standard heat treatment at 1050° C. is critically dependent on composition. As a result of the closer control possible, a considerably higher level of permeability in nickel-iron alloys produced by powder metallurgy can be ensured, compared with that attainable by conventional methods.

The development of initial permeability depends on the final heat treatment. In melted alloys it is also dependent on the purity of the hydrogen atmosphere in which the treatment is carried out and for optimum results it is essential to use exceedingly dry hydrogen. However, it has been found that with alloys produced by powder metallurgy this last factor is far less critical, with the advantage that such alloys can be heat treated in

* E. V. Walker, D. K. Worn and R. E. S. Walters. "Application of Powder Metallurgy to the Production of High Permeability Magnetic Alloy Strip." Iron and Steel Institute, Powder Metallurgy Symposium, December, 1954, Group III, pp. 56–65.

hydrogen with a dew point as high as 20° C. without any detrimental effect on the magnetic properties.

Table V compares the permeabilities determined with samples of strip produced by powder metallurgy with those of normal commercial alloys. The tests were carried out on strip 0·002 in. thick at a frequency of 2 kc/sec.

A further important characteristic of magnetic nickel-iron alloys produced by powder metallurgy is that they are less susceptible to certain " surface effects " which occur with the normal alloys causing the latter to show a heavy fall in permeability with decrease in strip thickness. The magnetic properties in powder metallurgy alloys can be fully retained in thin strip sizes down to 0·001 in. and 0·0005 in. thick. With an alloy of the composition ; Ni 77%, Fe 14%, Mo 4%, and Cu 5%, produced by powder metallurgy, it is possible to work to an initial permeability of at least 25,000 down to a thickness of 0·0005 in.

Further work is continuing on the subject of heat treatment, and it has been found possible, by suitable methods, to obtain an initial permeability of 60,000 : figures up to 100,000 have been recorded.

TABLE V.—INITIAL PERMEABILITIES OF STRIP.

| Sample | Initial Permeability | |
|---|---|---|
| | Annealed in Pure Hydrogen | Annealed in Wet Hydrogen |
| Powder-Metallurgy Alloys | 27,000–32,000 (mean 29,000) | 27,000–34,000 (mean 30,000) |
| Commercial Alloys | 18,000–33,000 (mean 28,000) | 13,000–20,000 (mean 17,000) |

The application of the powder metallurgy technique to the production of rectangular hysteresis loop alloys is being studied and preliminary results have shown some remarkable improvements, particularly with regard to the reduction of coercive force.

# Institute of Metals Spring Meeting in London

THE Spring Meeting of the Institute of Metals opened at the Royal Institution, Albemarle Street, London, W.1, on the evening of Tuesday, March 29th, when Professor F. C. Frank, O.B.E., F.R.S. (University of Bristol), delivered the Forty-fifth May Lecture on " The Nature of the Real Crystal." The meeting continued with technical sessions at Church House, Westminster, on Wednesday and Thursday, March 30th and 31st, and concluded with a series of visits to works in the London area on Friday, April 1st.

## Business Meeting

The official business of the Annual General Meeting of the Institute was dealt with on Wednesday morning, when the Report of the Council for the Year Ended 31st December, 1954, and the Report of the Honorary Treasurer and Accounts for the Financial Year Ended 30th June, 1954, were adopted.

*Officers for* 1955–56.—The Secretary then announced the names of members elected to fill vacancies in the Council for the year 1955–56, as follows :—

*President :* Dr. Maurice Cook (Joint Managing Director, Imperial Chemical Industries, Ltd., Metals Division, Birmingham).

*Vice-Presidents :* Mr. E. H. Jones (Joint Managing Director, Capper, Pass and Son, Ltd., Bristol) ; Mr. W. J. Thomas (Joint Managing Director, The British Aluminium Co., Ltd., London).

*Honorary Treasurer :* Mr. J. C. Colquhoun, M.B.E. (Chairman and Managing Director, The Manganese Bronze and Brass Co., Ltd., London).

*Ordinary Members of Council :* Mr. L. E. Benson (Metallurgist, Metropolitan-Vickers Electrical Co., Ltd., Manchester) ; Mr. C. F. J. Francis-Carter, O.B.E. (General Manager, Aluminium Wire and Cable Co., Ltd., Swansea) ; Mr. D. P. C. Neave (Managing Director, Imperial Smelting Co., Ltd., London) ; Professor H. O'Neill (Professor of Metallurgy, University College, Swansea).

*Senior Vice-President.*—The Council has elected Major C. J. P. Ball, D.S.O., M.C. (Chairman, Magnesium Elektron, Ltd., London) to be Senior Vice-President for the year 1955–56, and he will be the Council's next nominee for the Presidency of the Institute.

Following a vote of thanks to retiring Officers, the new President of the Institute, Dr. Maurice Cook, was invested with the Presidential Badge and inducted into the Chair. Dr. Cook then delivered his Presidential Address, in which he dealt with some aspects of progress in the non-ferrous field since the end of the first World War.

*Award of Medals and Prizes.*—The Secretary announced that the Council had made awards of medals and prizes as follows :—

*The Institute of Metals (Platinum) Medal for* 1955 to Dr. C. J. Smithells, M.C. (Past President), Director of Research, The British Aluminium Co., Ltd., in recognition of his services to metallurgical science, to the metal industries, and to the metallurgical profession.

*The Rosenhain Medal for* 1955 to Dr. W. A. Baker (Member of Council), Research Manager, The British Non-Ferrous Metals Research Association, London, in recognition of his outstanding contributions to knowledge in the field of physical metallurgy, with special reference to the influence of gases and shrinkage on the soundness of cast metals.

*The W. H. A. Robertson Medal and Premium for* 1954 to Professor Hugh Ford and Mr. J. G. Wistreich for their paper on " Problems of the Control of Dimension, Shape, and Finish in the Rolling of Sheet and Strip and in the Drawing of Wire " (*Journal*, 1954, March).

*Students' Essay Prizes for* 1954. First Prize to Dr. A. P. Miodownik (Student Member), Research Investigator, Battersea College of Technology, London, for an essay on " The Significance of Microhardness Testing," which has been recommended for publication in the Institute's " Bulletin " ; Second Prize to Mr. Surindar Singh (Student Member), Student, Wednesbury County Technical College, for an essay on " The Kirkendall Effect."

The Presentation of medals and prizes was made by Dr. Cook*, following his Presidential Address, which is reported elsewhere in this issue.

## Technical Sessions

Technical sessions for the presentation and discussion of papers were held on Wednesday afternoon and Thursday morning and afternoon. Two concurrent technical sessions were held each day, one of the Thursday programmes taking the form of an all-day symposium on " The Control of Quality in the Production of Wrought Non-Ferrous Metals and Alloys." Part III : " The Control of Quality in Heat-Treatment and Final Operations." The papers presented at the various sessions are listed below.

*Wednesday—Programme A*

*Theme : Refractories for Low-Frequency Induction Furnaces.*

Jointly : Thackwell, " The Choice and Construction of

* As Dr. Smithells was in the U.S.A. at the time of the Spring Meeting, the medal will be presented to him at the Autumn Meeting.

Monolithic Linings for Twin-Bath Induction Furnaces for Melting Aluminium Alloys"; COOK, COWLEY and BROADFIELD, "The Use of Refractories in Low-Frequency Induction Furnaces for Melting Copper Alloys."

*Wednesday—Programme B*

*Theme: Fatigue.* Jointly: FORSYTH, "Some Further Observations on the Fatigue Process in Pure Aluminium"; HANSTOCK, "Fatigue Phenomena in High-Strength Aluminium Alloys"; MCKEOWN, MENDS, BALE and MICHAEL, "The Creep and Fatigue Properties of Some Wrought Complex Aluminium Bronzes"; FORSYTH and STUBBINGTON, "The Influence of Sub-Structure on the Slip Observed in Pure Aluminium and Aluminium Alloys When Subjected to Fatigue Stress"; MARTIN and SMITH, "The Effect of Internal Oxidation on the Fatigue Properties of Copper Alloys."

*Thursday—Programme A (Symposium)*

SHILLING, "Assessment of Quality of Wrought Products"; FIELD and SALTER, "The Control of Quality in Heat-Treatment and Final Operations in the Production of Rolled, Extruded, and Drawn Aluminium and Aluminium Alloys"; MILLER, "Heat Treatment and Finishing Operations in the Production of Copper and Aluminium Rod and Wire"; HYSEL and COLLIER, "The Control of Quality in the Heat-Treatment and Finishing of Copper and Copper-Base Alloys"; EDMUNDS and LLOYD, "The Production of Light-Alloy Drop-Forgings, Their Heat-Treatment, Inspection and Testing"; BETTERIDGE and COUND, "The Heat Treatment, Inspection and Testing of Wrought Nickel and Nickel Alloys."

*Thursday—Programme B*

(1) *Theme: The Constitution of Uranium Alloys.* Jointly: GROGAN and PLEASANCE, "A Survey of the Uranium-Nickel System" with an appendix by (MISS) WILLIAMS on "An X-Ray Examination of Some Uranium-Nickel Alloys"; FROST and MASKREY, "The System Uranium-Lead"; FROST, "The System Uranium-Mercury"; BROOK, WILLIAMS and (MRS.) SMITH, "Pseudo-Binary Phase Sections Between Laves Phases in Ternary Alloys of Uranium"; SUMMERS-SMITH, "The Constitution of Uranium-Zirconium Alloys."

(2) *Theme: Twinning.* Jointly: CHURCHMAN, "The Formation and Removal of Twins in Titanium During Deformation"; BILLIG, "Growth Twins in Crystals of Low Co-ordination Number"; WOOLLEY, "Twinning and Untwinning in Polycrystalline Magnesium."

(3) *Theme: Deformation and Fracture of Zinc.* Jointly: CAHN, (MISS) BEAR and BELL, "Some Observations on the Deformation of Zinc at High Temperatures"; GREENWOOD and QUARRELL, "The Cleavage and Fracture of Polycrystalline Zinc in Tension."

# Correspondence

## ALUMINIUM IN THE OIL INDUSTRY

The Editor, METALLURGIA.

Sir,

Thank you very much for your copy of Mr. Bigford's reply to my comments. I can only say that he has a somewhat different interpretation of factor of safety from what I have. Surely the "safety" consists in having some margin between the test pressure and the final bursting pressure, and this margin is far greater in steel that in the aluminium pipes which he mentions.

I entirely agree with him in that "it is wrong to consider only one physical value", but this is surely exactly what he is doing in taking the proof stress as the criterion for strength. It is because there is so little margin between proof stress and tensile strength in the pipe that he mentions, that it is in my opinion not comparable with steel of similar yield point.

Yours faithfully,

J. S. BLAIR,

Stewarts and Lloyds, Limited.

Corby, 22nd March, 1955.

The Editor, METALLURGIA

Sir,

Further to Dr. Blair's letter dated 22nd March, I wish to reiterate that the strength of the Specification B.S. HT.10 aluminium alloy line pipes is more than adequate to withstand the relevant service conditions, and this is indicated and substantiated in the data given in my original article printed in your February issue.

This statement is further borne out by the constantly increasing satisfactory service experience which is being gained with the use of aluminium pipe line, typical instances being the off-shore line in Penang, Malaya, and the twelve mile line of $8\frac{5}{8}$ in. O.D. $\times \frac{1}{4}$ in. wall thickness pipe for conveying high pressure natural gas at 500 lb./sq. in. at Corpus Christi, Texas. The aluminium pipe line employed was only one-third the weight of the comparable steel pipe, so that full advantage had been taken of the favourable strength/lightness ratio of aluminium over steel. Typical physical and chemical properties such as corrosion resistance to salt water or sulphur in crude oil, smoother inside walls—reducing friction—and lighter weight, illustrate the suitability of aluminium for pipe lines, well tubing and casing, and many other purposes.

Yours faithfully,

H. M. BIGFORD,

Development Metallurgist, T.I. Aluminium, Limited.

Birmingham, 19th April, 1955.

## Gas Carburising Lecture

ON Thursday, April 21st, over a hundred engineers and metallurgists, many of them associated with the motor car industry, were entertained to dinner at the Manor House Hotel, Leamington Spa, by Electric Resistance Furnace Co., Ltd., prior to listening to a talk on gas carburising and associated processes, given by Mr. N. Koebel, Chief Metallurgist and Director of Research to Lindberg Engineering Co., Chicago.—U.S. associates of Electric Resistance Furnace Company.

## Copper Sales

THE Board of Trade announce that they have decided to sell up to about 45,000 tons of electrolytic copper over the rest of this year. Arrangements for the sale of part of this have already been made with the Rhodesian producers and their sales agents, and it is proposed to enter into early discussions with the trade about the disposal of the remainder. The intention is that so far as practicable the copper will be delivered and priced at an even rate. This copper is from the trading stocks remaining in the Government's hands after private trading was restored in 1953. The copper already sold is of Rhodesian brands.

# A Practical Strain-Hardening Function

By E. Voce, Ph.D., M.Sc., F.I.M.

*The article outlines progress in connection with the exponential strain-hardening function put forward in 1948, and discusses its advantages over the well-known power function of Ludwik. After considerable strain there is generally a change of régime, consistent with the change in the mechanism of deformation reported by Cook and Richards. The relationship between stress and indentation hardness is reviewed in the light of the exponential function, and the nature of the fillet connecting the strain-hardening curve with the elastic line is indicated.*

FOR an endless variety of applications connected with the cold working of metals and alloys, it is useful to have a simple and reliable equation to express the strain-hardening relationships. Such an equation was put forward in 1948[1], and since that date the analysis of several hundred published curves has amply confirmed its validity; in no case has the equation failed to fit the data with great exactitude. The work agrees with that of Cook and Richards[2], among others, in confirming the existence of a second régime of strain-hardening, beginning after a moderate degree of deformation. The second régime has the same equation as the first, but the constants take different numerical values. The purpose of this article is to indicate some of the advantages which the equation in question holds over its better-known rival, the power function of Ludwik, and to give a few illustrative examples.

Broadly, strain-hardening is the effect of deformation on the resistance to further deformation. Modern usage expresses deformation in terms of the natural, or logarithmic, strain $\eta$ and defines the degree of strain-hardening as the true stress $S$ at which plastic deformation can just be maintained under uniaxial load.

## Comparison with the Power Function

The two equations which it is desired to compare are :

*The Power Function :*

$$S = a\ \eta^{\beta} \qquad (1)$$

where $a$ and $\beta$ are constants ;

*The Exponential Function :*

$$S = S_{\infty} - (S_{\infty} - S_0)\ \exp\ (-\frac{\eta}{\eta_c}) \qquad (2)$$

where $S_0$ is the threshold stress at which plastic deformation begins, $S_{\infty}$ is the asymptotic stress attained after severe deformation, and $\eta_c$ is the characteristic strain which determines the shape of the curve.

As far as can be ascertained, the power function is purely empirical, whereas the exponential equation can readily be deduced from a few credible assumptions[3]. Though these may, and probably do, represent an oversimplification, they give to the exponential equation a physical meaning which the power function conspicuously lacks.

The power function suggests that all materials become indefinitely strong after severe deformation, which is a travesty of common sense. More moderate in its demands upon credulity, the exponential equation fixes an upper limit to the degree of strain-hardening attainable.

The power function defines a straight line when plotted on double logarithmic co-ordinates, whereas experience shows that real stress-strain data, when so plotted, invariably give a curve shaped like an italicised integration sign. No more than the central portion is even approximately straight. Quite recently, Halstead, McCaughley and Markus[4] have demonstrated this fact with remarkable clarity, and have categorically denied the validity of the power function. On the other hand, a double logarithmic plot of the exponential function forms a curve of the correct shape, as indicated in Fig. 1.

As most readers will be aware, the maximum load point in tension is that at which the slope of the true stress-strain curve becomes numerically equal to the stress (see Note 1). This is true of all stress-strain curves, irrespective of their form, but for the power function it means that $a = S_m\ \eta_m^{-\eta_m}$ and $\beta = \eta_m$, where $S_m$ and $\eta_m$ are respectively the true stress and logarithmic strain at the maximum load point. While these

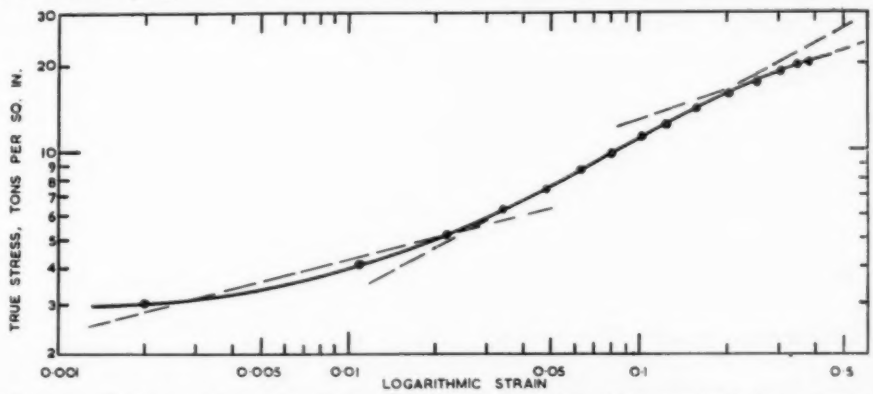


**Fig. 1.—Tensile true stress-strain curve for annealed high-conductivity copper, plotted on double logarithmic co-ordinates. The data points are from Table I, and the curve has been constructed from the exponential constants given in Table III.**

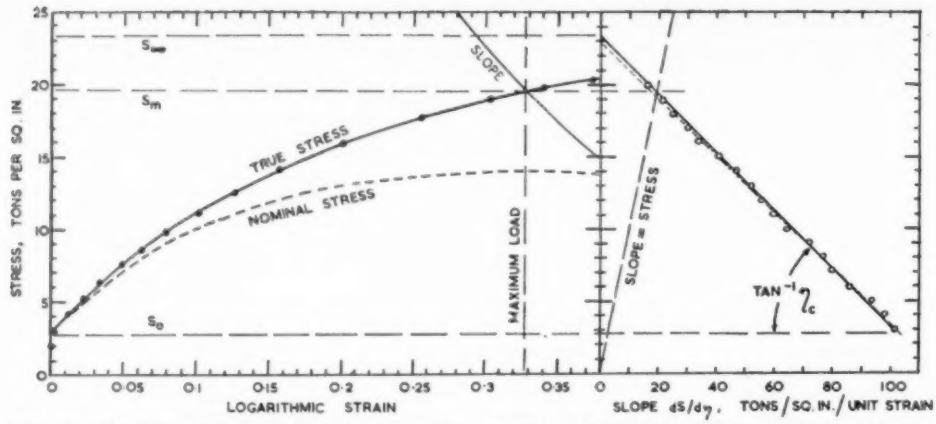


**Fig. 2.—Tensile true stress-strain curve for annealed high-conductivity copper, plotted on Cartesian co-ordinates. The curve is the same as that in Fig. 1. On the right of the diagram is the differential plot used to find tentative values of the exponential constants.**

identities lend to the constants a semblance of physical meaning, they show that the whole course of the curve is fixed by the co-ordinates of a single point upon it. We are faced with the absurdity that if several different materials happen to reach their respective maximum load points at the same stress and strain, they must necessarily have identical strain-hardening properties. In contrast, the exponential function imposes no such restriction, and different materials are free to reach the same maximum load point by different paths.

A necessary requirement of any acceptable strain-hardening function is that it should show the stress attained in a preliminary cold working operation to be that at which further plastic deformation begins. Only in this way can the function reflect the essential continuity of the strain-hardening process. Moreover, the logarithmic strains involved are, by their nature and definition, additive; they cannot legitimately be combined by multiplication (see Note 2). Re-arrangement of the exponential function gives

$$\eta = \eta_c \ln \frac{S_\infty - S_o}{S_\infty - S} \qquad (3)$$

It follows that, if $S_1$ and $\eta_1$ are, respectively, the stress and strain attained in a preliminary operation, then

$$\eta - \eta_1 = \eta_c \left[\ln \frac{S_\infty - S_o}{S_\infty - S} - \ln \frac{S_\infty - S_o}{S_\infty - S_1}\right]$$
$$= \eta_c \ln \frac{S_\infty - S_1}{S_\infty - S} \qquad (4)$$

Thus, the stress $S_1$ attained in the preliminary operation replaces the original threshold stress $S_o$, and becomes explicitly the stress at which subsequent deformation begins. The power function, treated similarly, leads to the comparatively meaningless expression

$$\eta - \eta_1 = \frac{1}{a}\left(S^{\frac{1}{\beta}} - S_1^{\frac{1}{\beta}}\right) \qquad (5)$$

This could only be consistent with the original equation if the right hand side were equal to

$$\frac{1}{a}(S - S_1)^{\frac{1}{\beta}}$$

which is untrue.

Furthermore, let us suppose that the power function holds for a material in the annealed condition, but that the only specimen available for test is one which has received a small unknown strain $\eta_1$, perhaps due to coiling or some similar process of mechanical handling in production. We shall then think that we are plotting our stress values against the total strain $\eta$ when, in fact, we are plotting them against $(\eta - \eta_1)$. If a double logarithmic plot gives a straight line against $\eta$, then it cannot do so when using the same stress values against $(\eta - \eta_1)$, and any attempt to draw a straight line through the plotted points must necessarily lead to erroneous values of the constants. In similar circumstances, the exponential function merely reveals an appropriately increased value of the threshold stress, with no change in the major constants, and no alteration in the shape of the curve. The effect is tantamount to moving the stress ordinate along the strain axis to a new position at $\eta_1$, using Cartesian paper. In essence, the exponential function is consistent and continuous with itself, when strains are combined additively, whereas the power function is not.

### Junction between Successive Regimes

Based on double logarithmic plots, French and Hibbard[5], as well as Jaoul[6], have reported the existence of two consecutive régimes of strain-hardening under the power function. In the diagrams of French and Hibbard this undoubtedly, and in those of Jaoul it probably, reflects an attempt to put a separate straight line through the lower part of the curve, where it sweeps backwards towards log $0 = -\infty$ at the threshold stress, as indicated by the lowermost broken line in Fig. 1. Nevertheless, changes of régime do, in fact, occur, and it is instructive to consider conditions at the transition point under the power and exponential functions respectively.

According to the power function, the slope of the curve at any point is

$$\frac{dS}{d\eta} = a\beta\ \eta^{\beta-1} = \frac{\beta S}{\eta} \qquad (6)$$

It follows that two régimes characterised by different values of $\beta$ cannot have the same slope at a point defined by any particular stress and strain. In other words, tangential junction is impossible, and any change of régime must be marked by an abrupt change of direction in the stress-strain curve. Though Jaoul

TABLE I.—TENSILE TEST ON ANNEALED HIGH-CONDUCTIVITY COPPER

| Load tons | Diameter in. | Sectional Area sq. in. | True Stress tons/sq. in. | Deformation Ratio* $A_o/A = \exp \eta$ | Logarithmic Strain $\eta = \ln (A_o/A)$ |
|---|---|---|---|---|---|
| 0·0 | 0·357 | 0·1001 | 0·00 | 1·000 | 0·000 |
| 0·2 | 0·357 | 0·1001 | 2·00 | 1·000 | 0·000 |
| 0·3 | 0·356$_5$ | 0·0996 | 3·01 | 1·002 | 0·002 |
| 0·4 | 0·355 | 0·0990 | 4·04 | 1·011 | 0·011 |
| 0·5 | 0·353 | 0·0979 | 5·11 | 1·022 | 0·022 |
| 0·6 | 0·350$_5$ | 0·0966 | 6·21 | 1·035 | 0·034 |
| 0·7 | 0·348 | 0·0951 | 7·35 | 1·050 | 0·049 |
| 0·8 | 0·346 | 0·0940 | 8·51 | 1·064 | 0·062 |
| 0·9 | 0·343 | 0·0924 | 9·74 | 1·082 | 0·079 |
| 1·0 | 0·339 | 0·0903 | 11·08 | 1·108 | 0·102 |
| 1·1 | 0·335 | 0·0881 | 12·48 | 1·135 | 0·127 |
| 1·2 | 0·330 | 0·0855 | 14·06 | 1·170 | 0·157 |
| 1·3 | 0·323 | 0·0819 | 15·88 | 1·222 | 0·201 |
| 1·37 | 0·314 | 0·0774 | 17·70 | 1·290 | 0·255 |
| 1·40† | 0·307 | 0·0740 | 18·92 | 1·352 | 0·302 |
| 1·40† | 0·301 | 0·0712 | 19·68 | 1·405 | 0·340 |
| 1·395 | 0·296 | 0·0688 | 20·28 | 1·454 | 0·374 |

* The deformation ratio diminished by unity is equal to the fractional increase in length.
† Ordinary tensile strength, 14·0 tons/sq. in.

appears to accept this conclusion, while French and Hibbard ignore it, sharp alterations of slope are certainly not commonly apparent in strain-hardening curves.

In contrast, the instantaneous slope for the exponential function is

$$\frac{dS}{d\eta} = \frac{S_\infty - S_o}{\eta_e} \exp\left(-\frac{\eta}{\eta_e}\right) = \frac{S_\infty - S}{\eta_e} \qquad (7)$$

If the constants for the second régime are $S_o'$, $S_\infty'$ and $\eta_e'$, then perfect tangential transition can, and does, take place at the stress $S_t$, where the slopes of the two branches become equal. This condition is defined by

$$\frac{S_\infty - S_t}{\eta_e} = \frac{S_\infty' - S_t}{\eta_e'}$$

whence the transition stress is

$$S_t = \frac{\eta_e' S_\infty - \eta_e S_\infty'}{\eta_e' - \eta_e} \qquad (8)$$

The corresponding transition strain $\eta_t$ can be found by putting $S_t$ into equation 3, using the first régime constants. The transition point is clearly the starting place of the second régime, and, in conformity with equation 4, the equation for the second régime is, consequently

$$\eta = \eta_t + \eta_e' \ln \frac{S_\infty' - S_t}{S_\infty' - S} \qquad (9)$$

It is, however, convenient to extrapolate the second régime backwards to zero strain by calculating the appropriate threshold stress $S_o'$ from a rearrangement of the basic equation, namely

$$S_o' = S_\infty' - (S_\infty' - S_t) \exp \frac{\eta_t}{\eta_e'} \qquad (10)$$

Though this leads to three constants for each régime, it is important to note that one of them is derived from the others. With six independent constants, the two branches, though parallel to each other at the transition point, would not necessarily touch.

To summarise the comparison, it can be said that the exponential equation is unquestionably preferable to the power function on every count which has been considered. The fact that the power function fails to cope rationally with the addition and subtraction of logarithmic strains, and, consequently, with partially strain-hardened materials, is alone sufficient to make it untenable as a practical strain-hardening function.

TABLE II.—ANALYSIS OF DATA IN TABLE I BY MEANS OF THE POWER FUNCTION.

(All stresses in tons/sq. in.)

| | First Régime | Second Régime | Third Régime |
|---|---|---|---|
| Primary Constants: | | | |
| Stress at Unit Strain .. .. $a$ | 13·6 | 38 | 27·5 |
| Index .. .. .. .. .. $\beta$ | 0·25 | 0·535 | 0·32 |
| Conditions at Maximum Load: | | | |
| True Stress .. .. .. .. $S_m$ | 9·6 | 27·1 | 19·1 |
| Logarithmic Strain .. .. $\eta_m$ | 0·25 | 0·535 | 0·32 |
| Deformation Ratio .. exp $\eta_m$ | 1·28 | 1·71 | 1·38 |
| Tensile Strength .. .. .. $T_m$ | 7·5 | 15·8 | 13·8 |
| Uniform Extension .. .. % | 28 | 71 | 27·4 |

## A Practical Example

Let us, however, examine the two equations in the light of an experiment. A tensile test on annealed electrolytically-refined high-conductivity tough-pitch copper was carried out on a lever-type testing machine, using Robertson grips to ensure axial loading. After each increment of load, measurements of the diameter were made at a number of positions along the gauge length. Until the onset of necking, when the test was discontinued, there was no significant variation of diameter along the gauge length, but the average value of the readings at each stage was used to calculate the sectional area. The applied load divided by the corresponding sectional area gives the instantaneous true stress, while the true strain is, by definition, the natural logarithm of the ratio of the original to the current sectional area. In the absence of necking, the area ratio is identical with the inverse length ratio, and the equivalent elongation is, therefore, available. The data are set out in Table I.

To express the results, even approximately, by means of the power function would require no less than three consecutive régimes, as indicated by the broken straight lines in the double logarithmic plot of Fig. 1. The values of the power function constants, namely, the stress at unit strain $a$, and the slope of the line $\beta$, are given in Table II. The middle régime is more representative of the test as a whole than either of the others, and it is unquestionably this straightish central portion of such curves which has beguiled authorities into acceptance of the power function. Yet its constants lead to completely erroneous results for the tensile strength of the material; only for the comparatively inconspicuous third régime are reasonably satisfactory values obtained.

Let us abandon the misleading double logarithmic co-ordinates, replot our data on ordinary graph paper, and examine the resulting true stress-strain curve in the light of the exponential function. Equation 7 can be expressed as the straight line.

$$S = S_\infty - \eta_e \left(\frac{dS}{d\eta}\right) \qquad (11)$$

It follows that if we plot the differential coefficient $dS/d\eta$ against the stress, we can find $S_\infty$ and $\eta_e$ from the intercept and slope respectively. Precise differentiation is quite unnecessary, for all we require at this stage are tentative values of the constants for trial; in any case, our free-hand curve through the plotted points is unlikely to be an exact mathematical function. A transparent protractor, pivoting about a needle, and a table of natural tangents gives ample accuracy, and a factor appropriate to the scales of plotting converts the tangents

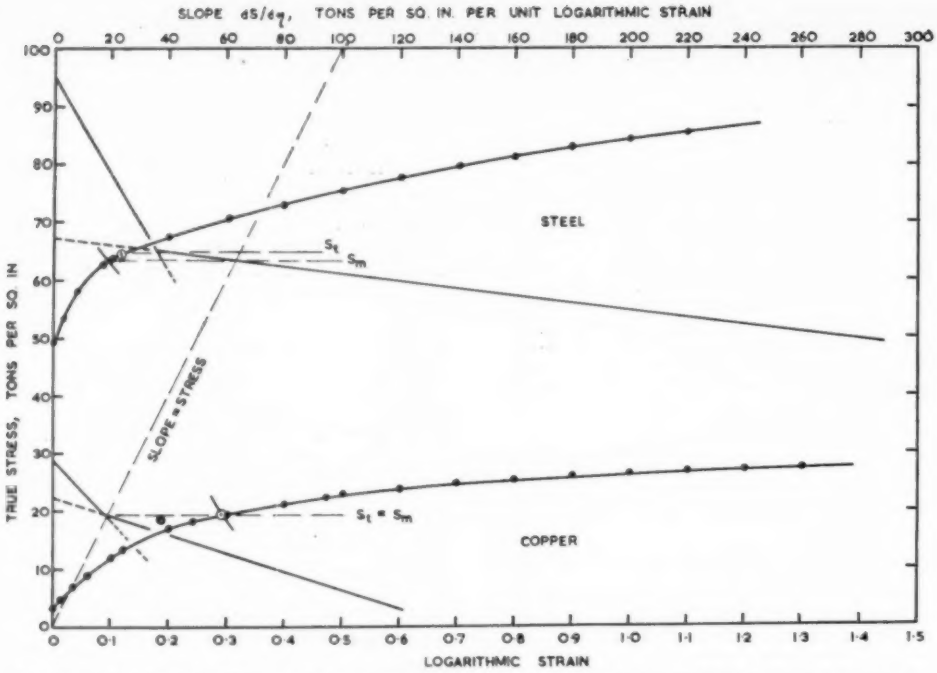
SLOPE dS/dη, TONS PER SQ. IN. PER UNIT LOGARITHMIC STRAIN
0 20 40 60 80 100 120 140 160 180 200 220 240 260 280 300
100 90 80 70 60 50 40 30 20 10 0
TRUE STRESS, TONS PER SQ. IN
STEEL
$S_t$
$S_m$
SLOPE = STRESS
$S_t = S_m$
COPPER
0 0·1 0·2 0·3 0·4 0·5 0·6 0·7 0·8 0·9 1·0 1·1 1·2 1·3 1·4 1·5
LOGARITHMIC STRAIN

**Fig. 3.—Tensile true stress-strain curves for copper and steel, corrected for radial stresses at the neck (Marshal and Shaw). The curves have been constructed from the exponential constants given in Table IV, and each shows two consecutive régimes of strain hardening. The differential plots derived from the same constants are superimposed on the diagram.**

to differential coefficients*. Fig. 2 shows the stress-strain curve together with the resulting differential plot. The latter suggested $S_\infty = 23$ tons/sq. in. and $\eta_c = 0{\cdot}20$, while inspection of the stress-strain curve gave $S_0 = 3$ tons/sq. in. After a few trials, an excellent fit was found by modifying these values to those recorded in Table III, from which the main curves in Figs. 1 and 2 have both been drawn. Thus a single sweep of the exponential function expresses accurately the information which the power function would require three ill-fitting régimes to convey.

### Conditions at the Maximum Load Point

Because of the equality of stress and slope at the maximum load point, it can be shown from equation 7 or 11 that

$$S_m = \frac{S_\infty}{1 + \eta_c} \qquad (12)$$

The corresponding strain $\eta_m$ can be found from equation 3, and the ordinary tensile strength $T_m$ follows at once from

$$T_m = S_m \exp(-\eta_m) \qquad (13)$$

The values computed in this way from the constants are included in Table III. The calculated tensile strength agrees exactly with that found by experiment, while the corresponding true stress and strain can be located with more precision than is practicable by experiment, because ductile materials like copper show a long, flat maximum on the load-extension diagram, and it is difficult to decide just where the critical point should be. The analysis pins it down to that at which the stress and slope are numerically equal. In the direct plot, as in the left hand part of Fig. 2, the critical point is that at which the stress-strain and slope-strain curves intersect, while in the differential plot, comprising the right hand part of the diagram, it is that at which the line representing slope = stress crosses the slope-stress line for the material.

### Examples and Nature of the Change of Regime

In the foregoing experiment, a single régime extends up to the maximum load point, where the radial stresses which accompany necking introduce a complication. Using a correction devised by Bridgman, however, Marshall and Shaw[7] have succeeded in extending the tensile stress-strain curves for copper and steel beyond the necking point without the interference of radial stresses. Their data are presented in Fig. 3, in which the curves have been drawn from the constants in Table IV. The tentative differential plots used to suggest trial values for the constants are not shown, but those based on the finally accepted values are superimposed on the

TABLE III.—ANALYSIS OF DATA IN TABLE I BY MEANS OF THE EXPONENTIAL FUNCTION.

(All stresses in tons/sq. in.)

| | | Calculated | By Experiment |
|---|---|---|---|
| Primary Constants: | | | |
| Threshold Stress | $S_0$ | 2·8 | |
| Asymptotic Stress | $S_\infty$ | 23·3 | |
| Characteristic Strain | $\eta_c$ | 0·195 | |
| Conditions at Maximum Load: | | | |
| True Stress | $S_m$ | 19·5 | 18·9–19·7 |
| Logarithmic Strain | $\eta_m$ | 0·328 | 0·302–0·340 |
| Tensile Strength | $T_m$ | 14·0 | 14·0 |
| Uniform Extension | % | 38·8 | 35·2–40·5 |

* It is quicker to plot the tangents directly against the stress, and to apply the scale factor to the apparent value of $\eta_c$ so obtained.

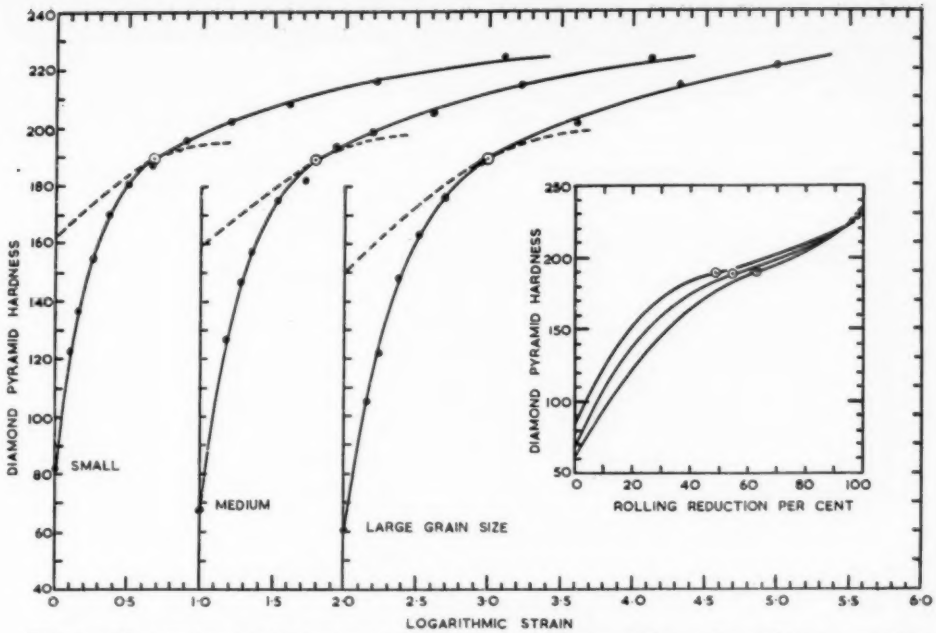


**Fig. 4.—Diamond pyramid hardness tests on 70 : 30 brass strip, cold rolled to various reductions. (Cook and Richards). The exponential constants from which the curves have been constructed are given in Table V. The change of régime coincides with the change in the mechanism of deformation described by the authors.**

diagram. Each is made up of two straight branches representing two successive régimes of strain-hardening. The branches intersect at the transition point where the slopes of the two régimes are equal. For the copper specimen, the transition point fortuitously coincides with the maximum load point, but for the steel the maximum load is appreciably exceeded before the second régime is entered.

Cook and Richards[2, 8, 9] have reported two successive stages in the mechanism of deformation by cold rolling. Referring to copper[8] they write: "During the cold rolling of polycrystalline copper up to about 50% reduction in thickness, the plastic flow can be accommodated by each individual crystal on its own set of 12 slip systems, and the process of slip on octahedral planes brings nearly all the crystals into orientations approximating to that of the final preferred orientation of the fibre textures. At this stage deformation can no longer take place by the normal slip mechanism, and the appearance of transverse striations across the grains indicates that the grains are breaking up into blocks by movement upon the flow layers. The flow layers are not coincident with any octahedral planes, and probably not even with any other important crystallographic planes. It seems probable, therefore, that after about 50% reduction, the mechanism of deformation consists of the movement or gliding of small blocks on layers of heavily distorted metal." In the case of 70 : 30 brass[9], they correlated similar changes of mechanism with diamond pyramid hardness values, and state that: "The evidence of X-ray and microscopic examination makes it clear that the deformation of 70 : 30 brass during progressively increasing cold-rolling reductions takes place by two mechanisms similar to those already described for copper." Referring to the curves for hardness against logarithmic rolling strain, they say: "The curves can each be resolved into two almost linear portions—namely, a steep first portion with rolling reductions up to about 50%, and a less steep second portion above 50%. This change of slope in the logarithmic work-hardening curves corresponds with the observed change in the mechanism of deformation, namely the change from deformation by slip to deformation by crystal break-up."

TABLE IV.—TENSILE TESTS ON COPPER AND STEEL, CORRECTED FOR RADIAL STRESSES AT NECK. (Marshall and Shaw).

(All stresses in tons per sq. in.)

| | | Copper | Steel |
|---|---|---|---|
| First Régime: | | | |
| Threshold Stress | $S_0$ | 2·9 | 49·1 |
| Asymptotic Stress | $S_\infty$ | 22·1 | 67·0 |
| Characteristic Strain | $\eta_0$ | 0·158 | 0·062 |
| Second Régime | | | |
| Threshold Stress | $S_0'$ | 11·5 | 59·8 |
| Asymptotic Stress | $S_\infty'$ | 28·8 | 95·4 |
| Characteristic Strain | $\eta_0'$ | 0·51 | 0·85 |
| Transition Point: | | | |
| Transition Stress | $S_t$ | 19·05 | 64·8 |
| Transition Strain | $\eta_t$ | 0·292 | 0·127 |
| Maximum Load Point: | | | |
| Critical Stress | $S_m$ | 19·05 | 63·0 |
| Critical Strain | $\eta_m$ | 0·292 | 0·095 |
| Tensile Strength | $T_m$ | 14·2 | 57·3 |
| Uniform Extension | % | 33·8 | 9·9 |

The hardness data in question are plotted against logarithmic rolling strain in Fig. 4, in which the curves have been drawn in accordance with the exponential equation from the constants in Table V. It is evident that the fit of the equation, under two successive régimes, is well within the experimental scatter of the hardness determinations. Though neither régime could be described as linear, the results are otherwise entirely consistent with the observations of Cook and Richards quoted above. Material of three different grain sizes was used, and the transition strain, always of the correct

order, was found to increase progressively with the grain size. This seems consistent with the conclusion that the first stage deformation is the result of slip within the individual crystals, for it is likely that larger crystals could accommodate more deformation than those of smaller size before becoming blocked. Moreover, the conception of the eventual cessation of slip is in excellent accord with the existence of an upper limit to the first régime of strain hardening, and so lends reality to the asymptotic stress or hardness.

Because rolling is more akin to plane strain than to simple tension, it is probable that the rolling strains recorded in these experiments should be modified by a constant factor. This would merely alter the numerical values of all the characteristic strains proportionately without affecting the argument.

Experience shows that the second régime constants are much more sensitive to the externally imposed experimental conditions than those of the first régime. For reasons beyond the scope of this article, it is believed that any acceleration or deceleration in the speed of deformation has an important effect on their numerical values, and may in some cases cause break-down of the exponential relationship. It is difficult to avoid local changes of speed in cold working processes, especially at large strains, where the rate of strain hardening, $dS/d\eta$, is low.

## The Relationship between Stress and Hardness

The results just described demonstrate that the exponential function can be applied to hardness tests on cold worked material as well as to stress-strain relationships, a fact which was first pointed out by Palm[10]. The relationship between stress and hardness was analysed in the light of the exponential function when discussing the work of Tabor[11, 12]. In essence, after allowing for the strain required to make the impression, the pressure on the indenting tool becomes directly proportional to the yield stress of the material, as, indeed, it is required to be by plastic theory. For cones and pyramids, like that of the Vickers test, the hardness number is itself a direct measure of the pressure on the indenter, though this is not the case for balls. When pyramid hardness numbers are plotted directly against true stress for the same degree of cold work, the result is a straight line which makes a positive intercept $h$ on the hardness axis, and which extends at least throughout the first régime of strain-hardening. In such circumstances, it can readily be shown that the characteristic strain must necessarily be the same for the hardness as for the stress tests, and that the indentation strain $\eta_i$ retains the constant value

$$\eta_i = \eta_o \ln \frac{H_\infty}{H_\infty - h} \qquad (14)$$

where $H_\infty$ is the asymptotic hardness. Since the observed hardness numbers refer to material strain-hardened by this amount in excess of the yield stress values, a correction must be applied by taking

$$H = H_\infty - (H_\infty - H_o) .\exp\ (-\frac{\eta - \eta_i}{\eta_o}) \qquad (15)$$

where $H_o$ is the threshold hardness. When this is done, the intercept disappears, and the hardness numbers (pressure on the indenter) become directly proportional to the stress.

In theory, the same relationship holds for the second régime, and, by moving the axes of reference to the transition point, it can be shown that the indentation strain remains unchanged. Unfortunately, however, experimental procedure has so much influence on the second régime constants, that only under very carefully controlled conditions is the second régime characteristic strain the same for hardness as for stress. Consequently the experimental stress-hardness relationship often degenerates into a curve at large deformations.

TABLE V.—DIAMOND PYRAMID HARDNESS OF COLD ROLLED 70:30 BRASS STRIP.

(Cook and Richards).

| Initial Grain Size (mm.) | | 0·01–0·015 | 0·025 | 0·06–0·065 |
|---|---|---|---|---|
| First Régime: | | | | |
| Threshold Hardness | $H_o$ | 82 | 67 | 60 |
| Asymptotic Hardness | $H_\infty$ | 195 | 197 | 200 |
| Characteristic Strain | $\eta_o$ | 0·235 | 0·29 | 0·39 |
| Second Régime: | | | | |
| Threshold Hardness | $H_o'$ | 162 | 159·5 | 150·5 |
| Asymptotic Hardness | $H_\infty'$ | 230 | 232 | 235 |
| Characteristic Strain | $\eta_o'$ | 1·35 | 1·47 | 1·63 |
| Transition Point: | | | | |
| Transition Hardness | $H_t$ | 188·5 | 188·5 | 189 |
| Transition Strain | $\eta_t$ | 0·672 | 0·790 | 0·993 |
| Rolling Reduction | % | 48·9 | 54·6 | 62·9 |
| Critical Point: | | | | |
| Critical Hardness | $H_m$ | 158 | 153 | 144 |
| Critical Strain | $\eta_m$ | 0·263 | 0·315 | 0·357 |

## The Elastic-Plastic Fillet

In the earlier publication[1], it was pointed out that the elastic line is connected to the strain-hardening curve by a short fillet which belongs properly to neither. Almost certainly the local distribution of stress is far from uniform, and some regions within the metal become plasticised while the rest remains elastic. Doubtless it is the gradual spread of the plasticising process through the metal which gives rise to the fillet.

Let us suppose that the stress which the elastic parts can support, i.e., the elastic limit, is $S_e$, while the plasticised parts can support a stress $S_p$ obtainable from the main strain hardening function. If a fraction $x$ of a representative sectional area $A$ has become plasticised, then the total load $P$ which the section can carry is

$$P = (1 - x)AS_e + xAS_p \qquad (16)$$

Dividing across by $A$ and solving for $x$,

$$x = \frac{S - S_e}{S_p - S_e} \qquad (17)$$

where $S$ is the actual stress on the section, namely $P/A$. At first the chance that any small increment of strain $d\eta$ will plasticise more material is high, for there are countless positions where it could do so. Later, however, when little material remains to be plasticised, the chance is greatly diminished ; in effect it is roughly proportional to the fraction remaining to be plasticised, so that

$$\frac{dx}{d\eta} = \frac{1 - x}{\eta_o''} \qquad (18)$$

where $\eta_o''$ is a constant of proportionality in logarithmic strain units. When $x$ is zero the strain is the elastic strain $\eta_e$ and integration therefore leads to

$$x = 1 - \exp\ (-\frac{\eta - \eta_e}{\eta_o''}) \qquad (19)$$

Combining equation 19 with equation 17 and rearranging,

$$\eta - \eta_e = \eta_o''.\ln \frac{S_p - S_e}{S_p - S} \qquad (20)$$

This is of the same form as equation 4, except that $S_p$

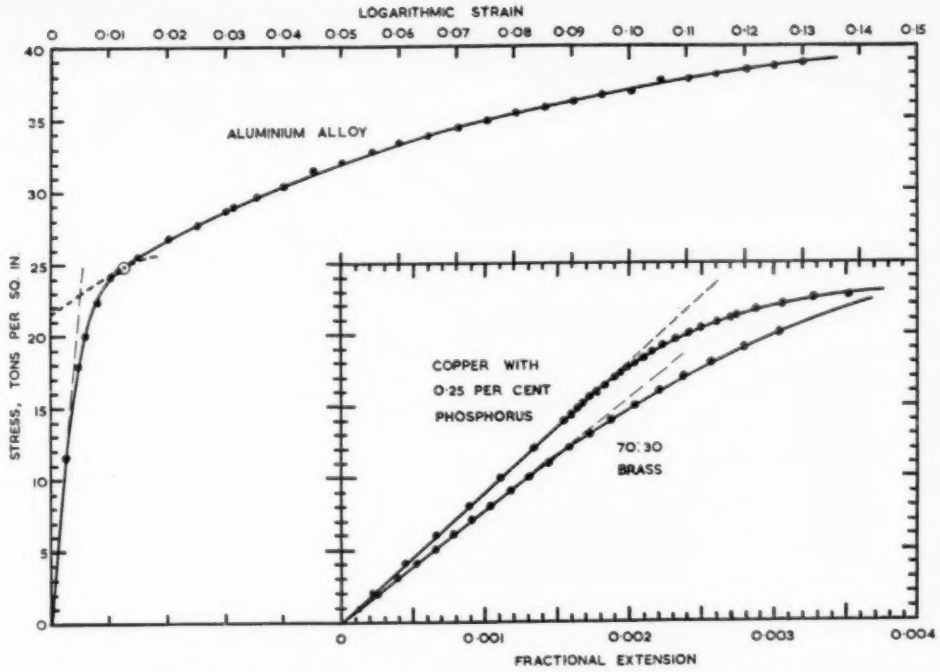
LOGARITHMIC STRAIN
0 0·01 0·02 0·03 0·04 0·05 0·06 0·07 0·08 0·09 0·10 0·11 0·12 0·13 0·14 0·15
STRESS, TONS PER SQ IN
40 35 30 25 20 15 10 5 0
ALUMINIUM ALLOY
COPPER WITH
0·25 PER CENT
PHOSPHORUS
70:30
BRASS
0 0·001 0·002 0·003 0·004
FRACTIONAL EXTENSION

**Fig. 5.—Tensile true stress-strain curve for an aluminium alloy (Marin), with other examples of the fillet connecting the strain hardening curve with the elastic line. The relevant constants appear in Table VI.**

unlike $S_\infty$ is a function of strain. For the small strains with which the fillet is concerned, however, $S_\infty$ changes very little, and can be replaced by a constant value $S_\infty''$ without appreciable error. This is slightly above the threshold stress of the main strain-hardening function, to allow for the small degree of strain-hardening involved. It follows that the fillet can generally be expressed by a brief preliminary régime of the exponential equation, with constants which permit tangential junction with the elastic line on the one hand, and with the main strain-hardening function on the other. Strictly, the fillet equation relates to true stresses and logarithmic strains, but the deformations are so small that nominal stresses and ordinary strains can be used if desired without introducing significant errors.

Though usually negligible in problems connected with the cold working of metals, these fillets are of considerable interest to engineers and designers, for the proof stresses, up to about 0·1% extension, generally lie upon them. Moreover, for light alloys and similar materials possessing comparatively little ductility, the fillet may comprise an appreciable part, or even the whole, of the total deformation before fracture occurs. Several examples are shown in Fig. 5, in which the curves are constructed from the constants in Table VI. The fillet for the aluminium alloy at the top of the diagram makes tangential junction with both the elastic line and the first régime curve, whereas the data for the other two examples are confined to the fillet and elastic extension alone. These are typical of extensometer tests for the determination of proof stress values; they refer to nominal stresses and orthodox strains, which, for the small deformations in question, are almost indistinguishable from true stresses and logarithmic strains. True stresses and logarithmic strains, were, however, used for the aluminium alloy, which represents the work of Marin[13].

## Determination of the Constants

The determination of the constants of an experimental curve should be regarded as a matter of trial and error, and with a little patience an almost perfect fit can usually be obtained. Four figure natural logarithms and exponentials give ample precision. The differential method of finding trial values of the constants has already been outlined. Alternatively, if $\eta_1$, $\eta_2$ and $\eta_3$ are three strains in arithmetical progression, all belonging to the same régime, while $S_1$, $S_2$ and $S_3$ are the corresponding stresses, then

TABLE VI.—EXAMPLES OF THE ELASTIC-PLASTIC FILLET.

(All stresses, slopes and moduli in tons per sq. in.)

| | Aluminium Alloy | Cold Worked Copper with 0·25% P | Cold Worked 70 : 30 Brass |
|---|---|---|---|
| Young's Modulus | 4460 | 8880 | 7725 |
| Limit of Proportionality | 11·6 | 15·9 | 10·0 |
| Elastic Strain | 0·0026 | 0·00177 | 0·0013 |
| **Constants for Fillet:** | | | |
| Threshold Stress | 11·6 | 15·9 | 10·0 |
| Asymptotic Stress | 25·5 | 23·8 | 32·0 |
| Characteristic Strain | 0·00311 | 0·00089 | 0·00285 |
| Initial Slope of Fillet | 4470 | 8880 | 7720 |
| **Strain Hardening Constants:** | | | |
| Threshold Stress | 21·6 | | |
| Asymptotic Stress | 42·1 | | |
| Characteristic Strain | 0·0733 | | |
| **End of Fillet:** | | | |
| Stress (transition) | 24·9 | | |
| Plastic Strain | 0·0098 | | |
| Elastic Strain | 0·0026 | | |
| Total Strain | 0·0124 | | |

$$S_\infty = \frac{S_2^2 - S_1 S_3}{2S_2 - (S_1 + S_3)} \quad (21)$$

$$\eta_o = \frac{\eta_2 - \eta_1}{\ln \dfrac{S_\infty - S_1}{S_\infty - S_2}} \quad (22)$$

$$S_o = S_\infty - (S_\infty - S_1) \exp \frac{\eta_1}{\eta_o} \quad (23)$$

Whichever method is used, it is generally desirable to compute the first régime threshold stress from the co-ordinates of a point well on the curve by means of equation 23, in order to avoid being misled by the fillet. The threshold stress for the second régime must invariably be computed in the same way from the co-ordinates of the transition point, as otherwise tangential junction will be lost.

## Conclusion

In this brief article, it has been possible merely to outline the value of the exponential equation as an eminently practical strain hardening function. Important advantages over the power function are :—

(1) that it fits experimental curves over longer ranges of strain ;
(2) that it can be derived inductively, in a manner very similar to that outlined above for the fillet ;
(3) that it sets an upper limit on the degree of hardening obtainable by cold deformation ;
(4) that it shows the stress attained in a preliminary strain-hardening operation to be *explicity* the threshold stress at which subsequent deformation begins ;
(5) that it copes rationally with the addition and subtraction of logarithmic strains, and consequently with partially strain-hardened materials ;
(6) that it permits different materials to reach the same maximum load by different paths ;
(7) that it provides for tangential junction between successive régimes ; and
(8) that it facilitates a satisfactory interpretation of the relationship between stress and indentation hardness.

Further examples of the application and theory of the exponential strain-hardening function are in preparation for publication.

## Notes

1. *Conditions at the Maximum Load Point*

The maximum load in tension invariably occurs at the point where the rate of strain hardening $dS/d\eta$ becomes numerically equal to the true stress. By definition, using the symbols in the text,

$$P = SA = SA_o \exp(-\eta)$$

where $A_o$ is the original sectional area. The load-strain curve reaches its maximum when

$$\frac{dP}{d\eta} = \frac{dS}{d\eta} A_o \exp(-\eta) - S\, A_o \exp(-\eta) = 0$$

That is to say, when

$$\frac{dS}{d\eta} = S$$

It follows that the maximum load point is also that at which the rate of work hardening, $dS/dW$, is unity. The work done per unit volume, $W$, is defined by

$$dW = S d\eta$$

Therefore, at the maximum load,

$$\frac{dS}{dW} = \frac{dS}{d\eta} \cdot \frac{d\eta}{dW} = \frac{S}{S} = 1$$

For such reasons, the true stress at the maximum load is a highly critical and important parameter. By means of equation 12, comparable critical points can be located on curves for compressive stress or hardness, although no actual maxima appear.

2. *The Combination of Strains*

If $A_o$, $A_1$ and $A_2$ are successive stages in the changing sectional area, then from the definition of logarithmic strain,

$$\eta_2 - \eta_1 = \ln \frac{A_o}{A_2} - \ln \frac{A_o}{A_1} = \ln \frac{A_1}{A_2}$$

Clearly the right hand side is the strain for the second stage alone, and the result is acceptable.

Similarly for deformation ratios,

$$\frac{A_1}{A_2} = \frac{A_o}{A_2} \cdot \frac{A_1}{A_o} = \exp(\eta_2) \cdot \exp(-\eta_1) = \exp(\eta_2 - \eta_1)$$

On the other hand, for fractional reductions of area,

$$\frac{A_o - A_2}{A_o} - \frac{A_o - A_1}{A_o} \neq \frac{A_1 - A_2}{A_1}$$

and similarly for fractional extensions (orthodox strains).

Thus logarithmic strains can be combined by addition and subtraction, and deformation ratios by multiplication and division, but neither orthodox strains nor fractional reductions can legitimately be combined in any comparable manner.


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## Cambridge Instrument Mechanical Thermometer Division

SINCE the recent establishment of their additional factory at North Finchley, and the consequent increased facilities available, the Cambridge Instrument Co., Ltd., have decided to concentrate there a complete self-contained unit to deal with their increasing business in mercury-in-steel indicating and recording thermometers ; vapour pressure dial thermometers ; automatic temperature regulators employing these systems ; and pressure and vacuum dial gauges and recorders.

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# Non-Ferrous Progress Since World War I

## Presidential Address to the Institute of Metals

*As one who has been intimately concerned for many years with the technical aspects of non-ferrous metallurgy, Dr. Maurice Cook devoted his Presidential Address to the Institute of Metals to an account of some of the important developments which have taken place in this field during the period of his membership of the Institute. A condensed version of Dr. Cook's address is presented here.*

DESPITE the lingering survival of some antiquated plants, the non-ferrous industry as a whole has undergone great changes in the space of a quarter-century, the increased use of electricity at all stages contributing, perhaps, more than any other single factor to this transformation. The scale and speed of operations have been increased and quickened. Highly mechanised plants have largely replaced simpler and smaller hand-operated units. Where appropriate, continuous processes are being introduced in place of batch methods, and, following the marked progress that has been made in instrument design, automatic control is being increasingly used. All these changes have raised production potential and efficiency, improved conditions of working, and eliminated much of the heavy manual labour that formerly and inevitably was associated with some processes. In addition to the advent of new manufacturing techniques and methods, the range of materials and products has been greatly extended to meet modern requirements.

### Melting and Casting

Going back more than 5,000 years, melting and casting are still the major primary operations in the non-ferrous metallurgical industry, and it is in this field that metallurgists have made some of their greatest contributions to industrial progress. With the advent of new alloys, craftsman's skill had to be implemented by techniques based on scientific practice.

Over the last thirty or forty years, metal gas reactions have been the subject of much successful research. Conspicuous in studies of gas equilibrium with copper has been the work of Allen and Hewitt on the combined effect of oxygen and hydrogen, and of Floe and Chipman on the role of sulphur. Investigations at the British Non-Ferrous Metals Research Association have shown that, in alloys not containing dissolved oxygen, solution and emission of hydrogen is the major source of unsoundness. In 1925, Portevin showed that additions of an oxidising agent could eliminate unsoundness in gun metal, and Lepp later proposed the use of oxidising slags as a means of controlling dissolved gases in copper alloys generally. That hydrogen was the cause of unsoundness in aluminium alloy castings was recognised in 1929 by Claus, who showed that it originated from decomposition of water by molten aluminium, a finding confirmed in 1931 by Hanson and Slater, who, amongst others, demonstrated the efficiency of treatments with chlorine or nitrogen for hydrogen removal. Realisation of the ill-effects of gases led to the accelerated adoption of electric melting.

The introduction of the copper-faced water-cooled mould, of the kind devised by Junker around 1926, has been responsible for a considerable improvement in ingot quality, and has facilitated the production of large ingots for modern plants. The use of continuous casting is another outstanding innovation, and the method originated by Junghans, some twenty-five years ago, has been successfully applied to the casting of brass and, more recently, copper. In 1936, German investigations showed that by using a short mould and effecting most of the cooling of aluminium alloy ingots by the direct application of water to the solidified skin, a greater degree of chilling, and hence better dispersal of phases, could be obtained than by conventional casting methods Such semi-continuous methods are now standard practice and are beginning to be used for copper and its alloys.

Fifteen years ago small buttons of high melting point metals were produced by arc melting on a water-cooled copper hearth, in vacuo or in an inert atmosphere, such as argon. Furnaces of this type are now in operation for producing ingots up to 2 tons, the solid metal being built up by lowering the hearth or raising the electrode as more material is introduced to the arc.

### Rolling

In general it can be said that the rolling of long lengths of coil in wide widths reflects the chief difference in sheet and strip rolling practice today compared with that of thirty years ago, when narrow coils of small weight were produced by slow speed rolling in two-high mills.

Since the 1920's, two-high mills have been altered out of all recognition by the incorporation of improved mechanical features and items of ancillary equipment to facilitate their efficient operation, combined with greater accuracy of construction. In the last forty years the speed of cold rolling strip mills has increased from 70 ft./min. to 1,000 ft./min., and even 2,000–4,000 ft./min. is not uncommon.

It is the four-high mill that has marked the greatest change in rolling installations. As long ago as 1926, a four-stand tandem mill of this type was erected in the U.S.A. for cold rolling 36 in. wide copper strip. A few years later, four-high mills were erected in this country, and for the hot and cold rolling of aluminium in widths up to 100 in., mass production methods have been adopted to an increasing extent on lines similar to those in modern continuous steel plants.

Other types of multi-high mills recently introduced include the Steckel mill, in which all the energy of rolling is transmitted by the coiler and decoiler; the Sendzimir mill, with work rolls as small as $\frac{1}{4}$ in. in diameter, and, for hot rolling, the planetary mill, in which in one pass it is possible to obtain a greater reduction than can be achieved in ten passes in conventional mills.

## Tube Production

The most radical changes in tube manufacture in recent years concern drawing operations. The first major change from single hole drawing on mandrels in short lengths (20–30 ft.) at a speed rarely exceeding 100 ft./min., was the introduction in the early 1930's of fixed plug drawing, which made possible the processing of longer lengths at higher speeds, and the drawing of several tubes simultaneously. Further development of this method, and of the floating plug technique and multiple drawing, led to the appearance just before the war of fully automatic triple draw benches, followed more recently by highly mechanised triple draw benches with capacities up to 300,000 lb. pull, and able to draw tubes up to 150 ft. long at 350 ft./min.

The work on plug design which made it possible to dispense with the plug rod and allow the plug to float, opened the way for block drawing, in which tubing, reduced to 2 in. diameter or less by conventional methods, is further drawn on bull blocks, coiled after each draw, and left in long coils or cut to length. This method has made possible the production of tubing in long lengths at 1,800 ft./min. Another departure from conventional drawing is tube reducing, which was introduced about 1930 and is particularly suitable for relatively hard alloys.

A substantial proportion of steel tubing is welded, but it is only recently that techniques for the production of non-ferrous tubes by welding have been devised.

## Extrusion and Wire Drawing

Markedly accelerated by the expanding use of light alloys, there has been a considerable growth in output of extruded materials, and among the interesting modifications in extrusion practice are the use of more durable materials for dies, mandrels, etc.; the introduction of glass as a lubricant; and the introduction of techniques for the production of hollow, tapered and stepped extrusions.

In the wire-drawing field, the extensive use of tungsten carbide dies and the replacement of single die units by high speed multiple-die and tandem machines are two examples of advances in plant design, operation and tool materials.

## Furnace Design

The open flame furnace long ago gave way to the muffle type, and the substitution of oil and gas for solid fuel facilitated improvements in quality and temperature control. The more general use of electricity led to the development of a wide variety of types of furnace, and associated with some of them has been the added development, started about twenty-five years ago, of a controlled atmosphere. Of other developments in thermal plant, the use of induction methods for pre-heating and heat treatment is amongst the most recent and interesting.

## Copper

In all types of copper alloys, new and improved materials have been produced in the past two or three decades, but attention will be directed to three sections of the copper alloy field—copper, condenser tube alloys, and precipitation-hardening alloys.

Copper has been available in high purity form for many years, and no less than 80% of the world production of primary copper is electrolytically refined. The contamination arising from the melting of cathode copper in reverberatory furnaces directed attention to the possibilities of arc melting, which was introduced on a large scale in 1936.

Until about 30 years ago most of the copper fabricated in wrought form was of the tough-pitch variety, and contained oxygen in amounts varying up to about 0·1%. When investigation revealed the role of oxygen in copper, not only in melting, but in contributing to cracking and distortion of the metal when heated under reducing conditions, and when welding as a means of construction started to make steady progress, the demand for deoxidised copper began. About 1930 techniques were developed for the production, without the use of a deoxidiser, of oxygen-free copper, which, while retaining the characteristic high conductivity of the metal, did not suffer the disadvantages of tough-pitch copper.

## Condenser Tubes

Ever since sea-going vessels have been fitted with steam condensers, the corrosion of condenser tubes has presented difficulties. Even as far back as 1890, the addition of 1% tin to 70/30 brass was made for service in more onerous conditions. For 45 years, first the Corrosion Committee of the Institute of Metals, and then, since 1930, the British Non-Ferrous Metals Research Association, has studied this problem, and it can fairly be claimed that work done in this country, including that carried out by industrial concerns, has been mainly responsible for solving the major problems of condenser tube corrosion, and for the development of corrosion-resistant alloys which have since become so widely used throughout the world.

Condenser tubes of 80/20 cupro-nickel were first made in this country in 1921, only to be superseded in 1925 by 70/30 cupro-nickel tubes, whose variability in performance led to the discovery of the improved corrosion resistance conferred by small amounts of iron and manganese. The British Non-Ferrous Metals Research Association extended its studies to the effects of iron on alloys of lower nickel content, and alloys with up to 10% nickel and 2% iron, with good corrosion resistance and capable of easy manipulation, have been developed for other sea-water carrying pipes in ships and power stations.

It was at one time thought that aluminium in brass impaired the corrosion resistance of the alloy, but work carried out at Woolwich for the B.N.F.M.R.A. in the early 1920's, and later in the Association's laboratories, showed the contrary to be the case, and an *alpha* alloy containing 2% aluminium was developed for condenser tubes. The presence of this amount of aluminium also greatly improves the resistance of brass to oxidation at elevated temperatures.

Corrosion which gives rise to dezincification in brass was at one time a serious trouble, but, around 1923, considerable variability of performance in service of brass condenser tubes was traced to the presence of arsenic. For the past thirty years it has been common, if not general, practice in this country to add 0·02–0·05% arsenic to *alpha* brass alloys for service where resistance to corrosion is an important consideration.

## Precipitation-Hardening Copper Alloys

Despite the use of copper alloys extending over thousands of years, nothing was heard of heat-treatable

copper alloys until the middle of the 1920's, when the investigations of Corson, Masing, Fraenkel and others revealed several such alloys, notably copper-beryllium. Since then, hundreds have been discovered, and a few have been used industrially to some extent.

The total amount of these alloys produced is very small in relation to other copper alloys, and whilst such special uses as there are for those heat-treatable alloys available in wrought forms do not at present involve the consumption of more than modest tonnages, the ever-increasing volume and variety of new developments in many fields suggests that this situation is one which could rapidly and appreciably change.

Knowledge of most of these alloys is very limited, and further investigation, of both a fundamental and technological kind, would appear to be merited. Further information about the nature of the hardening processes is needed, and the possibility of shortening the prolonged thermal treatments necessary to obtain optimum properties with some of these alloys is worth investigating.

## Aluminium

The greatest change in the pattern of wrought non-ferrous metal production in modern times is the three- or four-fold increase in the last fifteen years in output of semi-fabricated products in aluminium and its alloys, with which have been associated improvements in production technology of a most impressive kind.

One of the most important features in aluminium production since Hall and Heroult introduced their reduction process in 1886 has been the development of the three-layer refining cell for the production of aluminium of upwards of 99·99% purity from normal commercial purity material. From certain points of view the former has superior properties, and its use in making certain alloys would also seem likely to increase.

Although there seem to be large numbers of compositions in use, the principal aluminium alloys fall into six groups: three non-heat-treatable series—aluminium-manganese, aluminium-magnesium, and aluminium-silicon; and three heat-treatable types based on the aluminium-copper-magnesium-silicon, aluminium-zinc-magnesium, and aluminium-silicon-magnesium systems. The strongest of these—those containing zinc and magnesium—made their appearance about twenty years ago, and, along with the modified aluminium-silicon alloys, are amongst the most important developments in the period under review.

Cladding aluminium alloys with aluminium started only some thirty years ago, but it has been widely adopted all over the world, and has been extended to coating materials other than aluminium, such as aluminium-1% zinc alloy, which, being anodic to the core material, provides some measure of sacrificial protection.

## Effect of Small Amounts of Other Elements

The successes that have been achieved in the production of primary metals to high purity standards must certainly rank amongst the most outstanding advances that have been made in the past two or three decades. Usually corrosion resistance increases with the purity of a metal, magnesium providing a notable example, both as a parent metal and in alloys. The advent of high purity zinc in the middle twenties followed on the work of Hauer and Peirce, which showed that minute amounts of tin, lead and cadmium were responsible for the failure of zinc alloy die castings.

Until a few years ago it was usual in making strip and sheet in *alpha* brass alloys to cold roll throughout. With the need for processing much larger ingots into wider and longer coils, the attractiveness of initial hot rolling was clear enough, but only became possible and generally adopted by limiting the lead content to a maximum of 0·02%.

There are, of course, instances where the presence of small amounts of other elements improve the properties of a metal or alloy. Reference has already been made to the effect of small quantities of arsenic in inhibiting dezincification in *alpha* brass: more recently antimony and phosphorus have been found to behave similarly.

Another striking example is the vastly improved mechanical properties of the eutectic aluminium-silicon alloy brought about by the addition of small amounts of sodium. Much more recently, the beneficial effect of small additions of magnesium or cerium on the structure and properties of cast iron has been discovered.

## Titanium

The last few years have seen the advent of titanium as an industrial metal, and the technical achievements which have made this possible are amongst the most interesting of the period under review.

For 135 years, the properties of titanium were masked by impurities such as oxygen and nitrogen, but in 1925, van Arkel and de Boer produced metal of about 99·9% purity which was ductile and readily workable in both the hot and cold states. The development of a less costly method of extraction was left to Kroll, who, in 1940, was granted a patent for the production of titanium by reduction of the tetra-iodide with metallic magnesium, with subsequent removal of the residual magnesium and magnesium chloride by leaching. This pioneer work was continued in the U.S.A. under the auspices of the Bureau of Mines, and details of the various techniques developed were made known in 1946. Industrial production started modestly with 15 tons in 1948. In 1954, output reached some 4,000 tons, and the capacity of existing and projected plant in the U.S.A. is reputed to be about 35,000 tons.

## Joining

The last twenty years have seen a vast amount of work carried out on the metallurgy of welding processes, and much progress has been made in the welding of non-ferrous metals. The advent during the war years of the argon-arc process, and later of the argon-shielded metal-arc process, provided a means whereby consistently sound strong joints could be produced in such materials as aluminium and copper-base alloys, and led to their greater use in welded form.

Although argon-arc welding has been the most outstanding new feature in non-ferrous welding practice in recent years, progress in resistance spot and seam welding has led to a greatly increased use of these techniques for joining aluminium alloys. In joining them by methods other than welding, the use of organic bonding agents resulting from the work of de Bruyne, and of brazing techniques devised from the investigations of Miller and others, are amongst the most important developments.

## Surface Treatment

Although it is the properties of metals which largely dictate their use for any particular purpose, it is a com-

mon practice, either for decorative or protective reasons, to coat them in some way.

Geuther first electro-deposited chromium from a chromic acid solution in 1856, but it was not until the 1920's that it was taken up industrially. Due not so much to improvements in the composition of the bath as to a proper understanding of the operational control necessary, chromium plating has progressed from a somewhat unreliable but attractive novelty thirty years ago to a process of industrial importance for decoration and protection, for hard surfacing, and for building up worn parts.

The development of anodising of aluminium parts has proceeded alongside that of chromium plating from the work of Bengough and Stuart on the chromic acid process in the early 1920's. Important though it is as a protection against corrosion, anodising with subsequent coloration by dyeing has developed extensively in recent years and become a most popular form of decorative finish. Electro-deposited chromium and anodic oxide films have in common three major uses—protection against corrosion, decorative finishing, and hard surfacing.

Another form of protection, principally for ferrous materials against corrosion, is metal spraying. Zinc was one of the first metals to be tried, and is still widely used, but within the last fifteen years or so aluminium coatings have in turn been investigated, appreciated, and developed.

Tinplate is essentially a ferrous product, but it accounts for about half the annual consumption of tin, and its output, which runs into millions of tons a year, is produced in some of the most imposing plant employed in the metallurgical industry. Moreover, the accomplishment of implementing, within one decade, research work on electro-tinning into a continuous industrial process on a vast scale, must surely rank as one of the most striking metallurgical successes of our time.

## Analysis

Analytical control has always been a necessary and important feature in the wrought non-ferrous metals industry, and today is more important than ever. Effective analytical control is indispensable to many of the more recently developed processing techniques, and the success that has attended the efforts to develop rapid and accurate analytical procedures represents an outstandingly valuable contribution to the progress of the industry. Thirty years ago, the final determination was made gravimetrically or volumetrically, but today polarographs, absorptiometers and spectrographs are normal items of laboratory equipment.

In colorimetric techniques, tintometers have given place to absorptiometers, whose accuracy has been further improved by employing mercury vapour discharges, monochromators and spectrophotometers, as well as by general advances in electronic design.

The spectrograph has been by far the most useful of the various physical methods that have been devised, for its successful application in non-ferrous analysis has made it possible for results to be obtained in minutes rather than hours. This field has provided a notable example of highly successful co-operative research, carried out by the British Non-Ferrous Metals Research Association.

In the early years of their employment in industry, spectrographic methods were limited to the detection and estimation of small quantities by visual examination of spectra recorded on photographic plates: accurate measurement of line densities by micro-densitometer followed. The great advances in electronic design and components during the war led to a radical change in spectrographic procedure, and direct-reading spectrometers became available in the U.S.A. in 1945. As a class, they are certainly the most efficient analytical instruments so far developed, inasmuch as they provide for the accurate, rapid and economical estimation of most, if not all, metallic elements.

The results of continuing work on other methods, and current researches in X-ray fluorescence, mass spectrometry and radioactivity, indicate that the range of physical methods for analytical control may well be usefully extended in the future.

## Inspection and Testing

Modern production techniques, and the exacting nature of the conditions which some metals are required to withstand in many uses today, have resulted in a demand for new inspection and testing techniques.

Foremost among these is radiography, and the applicability of the technique to greater thicknesses of metal than can be penetrated by X-rays has recently been made possible by recourse to radioactive isotope sources, the most powerful so far available being Cobalt 60. For detecting deep-seated defects, ultrasonic methods, although not always so informative, have proved a useful alternative for examining large masses of metal which require longer exposure times by radiographic techniques. Another method of flaw detection which is making progress is the eddy current technique, which can also be applied to the assessment of internal conditions in tubing and the measurement of the thickness of strip and sheet.

The large increase, in the past quarter century, of processes and operations in which metals are used at elevated temperatures has necessitated the development of special mechanical testing techniques to determine the behaviour of metals under stress for long periods at such temperatures. In the same way, although as far back as 1864 Fairbairn studied the failure of riveted wrought iron girders subjected to repeated stress, it was the increasingly onerous conditions which metals were called upon to withstand in aircraft and other equipment during the first world war that gave rise to the intensive study of fatigue, which commenced in the early twenties and still continues.

## Conclusion

During the thirty-six years that have come and gone since the end of the first world war, our knowledge of metals has been vastly increased and profoundly deepened. As a result of this, and the immense strides forward that have been made in production technology, in improving processes and products, and in developing new ones, the industry has grown greatly, both in size and importance. Impressive as all these achievements have undoubtedly been, I think we can, with every confidence, look forward to still more outstanding advances in the future and the Institute, I am sure, can and will help importantly in their fulfilment.

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## Change of Name

We are advised that the name of the Royal Dutch Steelworks, Koninklijke Nederlandsche Staalfabricken v /J. M. de Muinch Keizer N.V., has been changed to Koninklijke Demka Staalfabricken N.V.

# The Training of Part-Time Metallurgical Students

By C. S. O'Dell

*In the author's view, the part-time metallurgical student has little opportunity of presenting his outlook on the matter of his training. In an attempt to remedy this state of affairs, he considers in this article the character of the part-time student, and the necessity for providing for him a definite training schedule. A training system designed to produce a keen, well-educated and thoroughly adaptable metallurgist is then discussed.*

A STUDY of the training of part-time students involves a consideration of the relationship, each to the others, of (*a*) the trainee, (*b*) the employer, and (*c*) the college. This can be represented pictorially as an equilateral triangle, as each is equally dependent on the other two, and should one fail in its duty the relationship becomes unbalanced.

Though this may appear obvious, it was, in the author's opinion, overlooked at the discussion on "The Training of Metallurgists for Industry," sponsored by the Institute of Metals and held at the Royal Institution, London, in November, 1953. The morning session was devoted to education in the universities, colleges, and technical schools, and the afternoon to training in industry. It was noticeable that everything was discussed at a high level;' lecturers, managers and senior metallurgists all had their say, yet nowhere was provision made for the trainee to present *his* outlook. Another point of note regarding the discussion was the lack of interest shown in the part-time student as compared with his full-time counterpart. For some reason the former seemed right out of favour, particularly with the employers, and this was a great disappointment.

It is because the viewpoint of the trainee has so seldom been given that this essay has been compiled. It is not an attempt to open the eyes of overworked managers, or even to create a lab-boys union, but rather to convey the requirements of training as envisaged by a part-time student.

## THE TRAINEE

### His Character

The part-time and evening student is a most enduring and ambitious person, because he is attempting to make a success of both work and studies at the same time, and is, therefore, fighting against the well-known saying that "No-one can properly do two jobs at once." This often means that, after a half or full day's work, the trainee may have to journey 15 or 20 miles for classes, and is quite willing to do so on two, or even three, nights a week, if he is convinced that his effort is worthwhile.

There are times, of course, when things do not go too well: when the trainee has an upset and is sure life would be far easier as a greengrocer's mate, with no travelling, no studies and little training. However, these matters invariably sort themselves out, and the ambition to become a fully-fledged metallurgist is restored to full vigour.

It is only through hard work that the part-time student can hope to achieve his ambition, for although statistics are not available, it is generally thought that the numbers qualifying by this method are decreasing, so things are not too hopeful for him from the beginning. However, if he is being helped at both his firm and college, and can feel that people are interested in him—that he is not fighting alone—then his ambition seems that much nearer.

The immense value a training scheme can be to serious junior metallurgists can therefore be appreciated. The trainee, becoming aware of an invisible link between company, college and himself, feels a sense of security, and becomes increasingly interested in both his work and his studies.

### Necessity for His Training

The majority of part-time students enter industry when 16 years old. They have received a secondary education which has given them a G.C.E. standard in physics, chemistry, mathematics, English, etc. This means they are reasonably well educated in basic subjects, though they know little or nothing of metallurgy. If they are to acquire this knowledge, then it must be jointly provided by the employer and the technical college, the former providing a practical bias and the latter a theoretical one. Through the years the part-time student is then brought to the level of his full-time colleague. This again shows the need for a definite training system.

## THE EMPLOYER

### Administration

It need hardly be stated that, although training schemes are the concern of all three parties, their actual administration is solely in the hands of the employer. Perhaps this is a good thing, perhaps it is not. Granted, it would be rather strange watching a trainee supervise his own training (though perhaps not so strange as watching industry subordinate to the commands of technical colleges), but the real drawback of such administration seems to be that, whereas one trainee can be compared with another, and one technical college with another, no two companies seem in any way alike. In one company there may be just an inspection laboratory measuring depths of case or checking hardness figures, whereas another may have five or six different laboratories, all engaged, of course, on Government research! However, though the sizes of companies, and the nature of their work are not comparable, it is thought that the structure of their training systems should be basically similar.

## Objective of Scheme

The primary objective of the training scheme should be to produce, upon his coming of age, a keen, well-educated and thoroughly adaptable metallurgist. This objective may well be considered as involving the provision of the following :—

(*a*) a stage-by-stage training programme ;
(*b*) a pleasant working atmosphere ; and
(*c*) incentives for hard study and work.

It is felt that if all parties keep these requirements in mind, the final objective will undoubtedly be reached, It is now necessary to examine each of the three in turn.

### *Stage-by-Stage Training Programme*

The first month or so in industry is the telling time, during which the trainee gradually changes from a schoolboy to a working man. It is then that he hopes to become interested in his work, and realises that if he does not do so during these first months it is unlikely that he ever will. Whatever happens, then, the trainee must be given worthwhile jobs, as his interest will not be sustained by " clearing out cupboards and fetching tea," nor, for that matter, by being a looker-on.

There are few things worse for the trainee than to watch his senior do everything ; it creates an impression that there is no confidence in him. To learn anything under these conditions, the trainee has to ask question after question, until the senior finally loses his patience, tells him to keep quiet, and thereafter looks upon him as an unwanted nuisance.

From the trainee's point of view, the best method of learning a process is by trial and error. First. there should be a brief discussion on the " whys and wherefores," and then the trainee should try the technique himself. On etching for the first time, the trainee may find the specimen to be " as black as night," even though it was agitated and timed to the nearest half-second just as the book said ; but after two or three faulty attempts the trainee realises that his own eyes are a far better guide than the stop-watch, so he decides to judge by eyesight on the fourth attempt. That the etch has ideal contrast is of little importance; compared with the fact that the trainee has thought for himself, and proved that it needed more than a reference book to deter him. He has probably taught himself more in that half-hour, than he would have learned watching someone else do it for a month.

It is almost certain that one of the first jobs to be taught will be the process of polishing microspecimens. Some trainees have it to do incessantly for the first three months—just polishing, polishing, polishing, without even seeing the specimen etched or under the microscope. Making polishing a drudge, instead of an essential metallurgical technique, is one of the major faults in some establishments. (After all, a metallurgist who cannot polish is about as useless as a footballer who cannot kick). A better method is to mingle the polishing with small jobs of responsibility, such as hardness checks or anything simple which the trainee can more or less complete himself.

There comes a time, usually after about three to six months, when the trainee desires his first posting. Until then he has merely been acquainting himself with elementary processes and equipment in general ; now he feels worthy of a place in a section. In a company that has more than one section or laboratory, a trainee will undoubtedly welcome the opportunity to move from one to another. Perhaps the best place to start is the inspection laboratory, where the jobs are usually small and varied, but, nevertheless, most important. The inspection departments have control of most of the fabrication and heat-treatment processes in industry, so that castings, tube-ends, and welds are but a few of the samples to be checked. The work on this section is of a practical nature rather than theoretical, and so is ideal for the first-year part-time student. It requires frequent use of the hardness tester and microscope, and so acquaints him with these and allied procedures. It is thought that on a section of this type a good general background to metallurgy may be obtained.

After about a year, it is recommended that a further move may be made, perhaps to the mechanical testing section. The trainee will no doubt feel that it is time he had a look at the more specialized branches of metallurgy, though he would still prefer to move from one branch to another at yearly intervals than be trained as a specialist. By moving round in such a manner the trainee can decide for himself which branch of metallurgy he wishes to specialise in upon completion of his training, as well as appreciate the relationship between one section and another. For instance, heat-treatment requirements are all the more understandable if training has been received in the mechanical testing section, than otherwise.

By moving from one section to another, the trainee obtains considerable general knowledge of metallurgical processes, which is considered preferable to receiving instruction in a specialised field. Particularly is this true of the part-time student, who likes to relate his work to his studies, as far as possible.

### *Pleasant Working Atmosphere*

This requirement can be divided into two distinct factors—human relationships and working conditions—the former depending on the people themselves, and the latter on the laboratory and its apparatus. Together they should provide conditions in which the trainee can spend a pleasant working day. Human relationships have always been, and will always be, of great importance in the training of metallurgists. This is because the relationship between the trainee and his immediate senior can be the making or breaking of the former's career. To have someone to confide in gives him a sense of security, and to be helpfully instructed makes the work far more interesting. Now and then, in an effort to make an impression, the trainee will slip up and make an unfortunate mistake. The senior should be prepared for this and have the necessary restraint so as not to lose his temper when such things happen.

The head of the department also has an important role to play, for he should be fully acquainted with each of his staff, both seniors and trainees, so as to be capable of settling any misunderstandings which may arise. The following story shows that some heads are unaware of this responsibility. After receiving two year's training in a firm, John Bradley found a better job, so one morning gave his notice to the secretary, who in turn handed it to the head. About half-an-hour later the head came through the laboratory asking which one was Bradley ! That he did not know his staff, even by name seems incredible. The atmosphere in this laboratory must be such that the trainee considers it a privilege if the head says ' Good morning '. Surely it would be far better if the latter spent ten minutes a month on an

encouraging chat with each trainee, for it is certain that both would benefit from the idea.

The question of working conditions depends greatly on the size of the company. Usually, the larger the company, the bigger the laboratories, and the more up-to-date the equipment. However, one does sometimes hear of companies with plenty of capital who do not believe in buying modern equipment, yet still expect first-class results. In such places equipment is mended and mended until it consists of little else than string, wood and plasticine. A disadvantage of such procedure from a trainee's point of view, is that because a firm refuses to purchase a simple piece of apparatus, he has to do the most ridiculous of jobs. Due to an automatic wire-cutter being refused in one laboratory, a trainee spent his days cutting wire into $\frac{1}{8}$-inch lengths 100g. at a time! The irony of this case was that just as the job was nearing completion the firm produced one made by themselves! Such conditions as these make the trainee most despondent, for he cannot produce first-class work with second-class materials, and if he is not allowed to make a good job of his work, then interest is soon lost.

Just as important as the quality of apparatus is the actual working environment. To produce a high-quality polish on specimens it is necessary to have a dust-free laboratory; to obtain accurate readings from instruments, it is necessary to have as much light as possible; and where calculations are made quietness is essential. Bench space is a requirement too, as it is far better to set apparatus out distinctly rather that jumble it together. All these considerations, together with many others, should be borne in mind if the trainee is to have an even chance of giving of his best to his employer.

*Incentives for Hard Work and Study*

The keenness of the part-time student has already been mentioned, but it is felt that the firms should be just as keen to see him get on. This can be achieved by providing incentives which urge the trainee to greater interest in his work.

Whatever happens, the progress of each trainee should be carefully assessed, and the best method of doing this is to appoint a trainee's supervisor. It is recommended that this person be someone in the capacity of senior metallurgist, rather than the head of the department, who is usually far too busy with other problems. The person appointed should have personal contact with each of the trainees, and be aware of the course each is taking at school, and the stage of training each has reached at work. The supervisor will be the 'voice of the firm' as far as the trainee is concerned, and any compliments or brickbats received will be given careful consideration. From him also, the trainee will learn the next step to which he can rise. By being told that if he passes his examination at the end of the year, and is also considered satisfactory at his present work, he will move up one position, the trainee knows exactly where he stands, and that from then onwards it is entirely up to him. Give the part-timer an aim and he will most certainly have a crack at it.

It is interesting to look at the following scheme, devised to help first-year part-time students understand their work. The supervisor realised that the S1 course included physics, chemistry and mathematics only, so that his trainees were receiving no instruction in metallurgy. A short course was run by himself to answer questions such as "Why heat-treat?" and "What happens during polishing?". An attempt was also made to show the relevance of the S1 course to the work. That this idea appealed to the trainees is shown by the fact that they were willing to give up part of their dinner hour twice a week to attend the course.

The question of wages is, of course, important, and, in connection with it, a point worth considering is the possibility of a definite wage structure. By definite is meant decisive as distinct from rigid. The trainee sees his pay-packet as an expression of his success at his training, and if he receives five shillings less than another he is naturally surprised and wishes to know why. Is it due to bad conduct or lack of output? If the supervisor has a scale at hand with everything 'cut and dried', there should be little reason for discontent.

At one firm the trainees who had been there for one or two years suddenly found that they were getting less than those who had just started. On being told, the departmental head got together with his senior assistants and drew up a wage scale by which all differences were soon settled. It consists of a simple table which recognises age, examination successes, and general conduct; so that each trainee knows exactly where he stands. For example, an 18-year old taking S2 may receive £12 per annum more than someone the same age taking S1. These small bonuses are of considerable help in stimulating interest in studies during the early years. If the scale is graded from S1 to A.I.M., the trainee can see just how worthwhile it is to qualify.

It is a common mistake by some firms to consider a reasonable wage as the only incentive a trainee needs. Though this is an important item, the trainee finds spoken encouragement far more stimulating.

## THE COLLEGES

It is interesting to note the relevance of these three requirements to the technical colleges. The stage-by-stage system of training is given as to be eligible for the next grade, failure meaning that the year must be repeated. It should also take the form of a small wage increase for each examination success.

The question of a pleasant working atmosphere again brings the problem of human relationships—this time those between the student and the lecturer. It is the duty of the latter to infuse interest in his subject, to present it as though it is the most exciting topic on which to talk. The majority of students agree that there are few things worse than listening to a tutor lecturing on a topic in which he has no interest. They also have no time for the lecturer who announces that quite a few people disagree with Smith's theory (and he's one of them), but it is in the syllabus and so must be learnt. From the student's point of view, the ideal lecturer is one who during theory talks "down to earth", and during practical work uses the personal touch.

## SUMMARY

It has already been hinted that at school-leaving age the student has probably never heard of a career in metallurgy. Perhaps the only reason he comes for interview is that he wants to work in a laboratory, and this seems a suitable sort of job. It will be seen that the supervisor must, therefore, be careful with his 'sales talk', as whether or not the applicant is attracted to the work is entirely dependent on him. It is useless taking him round the laboratories, saying this is the projection microscope, this is the tensometer, this is the electrolytic polisher, for these names will merely confuse him and put him off right away. On the other hand if the training system is explained, and he can see, and perhaps talk to trainees working under the system, he will naturally be more interested.

For the larger companies, a better method would be to issue a brochure explaining the objects and function of the training system. A copy could then be given to each applicant at the interview for further reading at home. Also, they could be sent to schools so that final year students become aware of the profession and the opportunities it offers.

Once attracted into the firm the trainee needs to be kept interested and contented. He will certainly do his utmost to achieve success at both studies and work and by following a training scheme similar to the one outlined here, the employer would be giving all the help required.

With all parties pulling their weight to this extent, the reward comes after six or seven years when a qualified metallurgist emerges who is a credit to himself, his college and his employer.


# The Protection of Structural Steel

THE Corrosion Group of the Society of Chemical Industry had a gratifyingly high attendance at their symposium on "The Protection of Structural Steel", held in London on March 31st and April 1st under the Chairmanship of Dr. J. C. Hudson of the British Iron and Steel Research Association. Well over two hundred engineers, steel makers, steel users, paint and metal finish manufacturers and academic workers attended, together with an impressive foreign contingent from a number of countries.

A wide variety of papers was delivered, followed by considerable discussion. Widespread interest was shown in the relative merits of paint and metal spraying protective techniques, particularly in the question of comparative costs. The testing methods employed in different countries were explained in detail by several of the foreign visitors, and the Society's Spring Lecture, delivered by Mr. F. L. La Que of The International Nickel Company, Inc., on U.S. activities in this field, was especially appreciated.

Mr. La Que traced the history of several technical groups in the United States that deal with corrosion to a major extent. Particular reference was made to the Inter-Society Corrosion Committee sponsored by the National Association of Corrosion Engineers, this latter organisation itself, the American Society for Testing Materials, the Electrochemical Society, the National Bureau of Standards and other governmental groups concerned with corrosion.

Mr. La Que also reported on the results of a recent survey of education in corrosion in American engineering schools. This showed, he said, that there was some instruction in corrosion in 37 schools and that 550 students were taking these courses. In addition to these specific courses, corrosion is dealt with in at least one course in 103 schools. There is activity in corrosion research in 34 institutions, with 91 students being engaged in these activities.

After the lecture a coloured sound motion picture covering activities at International Nickel's "Kure Beach" Corrosion Testing Station near Wilmington, North Carolina, was shown. Recognised as the most extensive research project of its kind in the world, this station embraces facilities for exposure of specimens to sea air at Kure Beach and salt water at neighbouring Harbor Island.

Mr. La Que's lecture was preceded by a pleasant ceremony at which he presented, on behalf of the National Association of Corrosion Engineers this year's Whitney Award to Dr. W. H. J. Vernon, O.B.E.

The Society's dinner at the Connaught Rooms was a happy function at which over one hundred guests entertained the foreign visitors. Several speakers stressed the value of the Symposium in providing a forum for both formal and informal exchange of views among workers in the same field. In proposing the toast of the Corrosion Group, Mr. J. M. Leonard, President-Elect of the Society of Chemical Industry, stressed the value of the Group's work in the four short years of its existance, and predicted for it a future of over-growing importance.

There can be little doubt that in bringing together the many personalities and interests involved in the important field of corrosion research, the Society of Chemical Industry is providing a service of vital importance to industry.

## Magnesium Elektron Ltd.

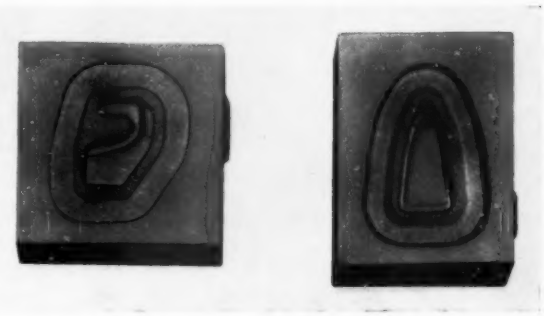
DISTILLERS CO., LTD., and the British Aluminium Co., Ltd., announce that the latter has acquired a majority interest in Magnesium Elektron, Ltd., previously a wholly-owned subsidiary of the Distillers Co., Ltd. The policy of M.E.L. will remain unchanged and the same high standard of co-operation between M.E.L. and their customers will be maintained. Major C. J. P. Ball remains Chairman of the Company, Lt.-Col. D. Lewis and Mr. C. P. Percy have resigned and the British Aluminium Co. have nominated the following to the Board of M.E.L.: Hon. C. Cunliffe (Deputy Chairman & Managing Director, B.A. Co.), Mr. W. J. Thomas (Managing Director, B.A. Co.), Mr. James Ritchie (Financial Director, B.A. Co.), Dr. C. J. Smithells, (Director of Research, B.A. Co.). This amalgamation will undoubtedly strengthen the position of the magnesium industry in the U.K. generally and in particular will strengthen the position of Magnesium Elektron, Ltd.

# Precision Alloy Steel Castings

## Wide Scope of B.S.A. Developments

NOT so very long ago, the prospect of using milling cutters that had been produced by a casting method seemed most remote. The very nature of ıstings, with their tendency to " brittleness ", seemed to e against the tremendous punishment that milling cutter eth had to withstand. In 1949, B.S.A. Tools, Ltd., erfected a process for the precision casting of milling ıtters, in which the resulting metallurgical structure ıd mechanical properties very closely resembled those f the conventional types of high-speed steel. The ımediate advantage then was a considerable reduction ı the cost of producing milling cutters—in the region of 0%. In addition to this, and of particular importance, as the fact that the casting process relied on the use of a teel in which chromium was the chief alloying element ıstead of tungsten or molybdenum, which were in very hort supply at that time. Of even greater value to the ool designer was the freedom of design that the casting rocess offered, since he was no longer restricted by onventional methods of machining from the solid. Staggered-tooth cutters of improved design and superior utting performance could be cast as easily as the straight tooth variety, and both could be produced more heaply than by conventional machining methods.

So rapid was the success of cast cutters, that the idea was soon conceived of expanding the process to include the precision casting of other articles, such as dies for forging, extrusion and plastic moulding, and general engineering components, where the saving of time and cost could benefit a much wider field. Considerable interest was aroused among customers, who co-operated during the initial development period, and it was soon evident that to exploit the process fully on a sound commercial basis, a separate company would have to be formed. In August last, Precision Alloy Castings (Birmingham), Ltd., officially came into being for the purpose of developing and applying the original B.S.A. Tools precision casting process to a much wider field. Already the new company is handling a large number of products for the jig, tool and die industry, and for the general engineering industries. Designers are rapidly revising their outlook towards casting as a technique, and this new precision approach has opened the door to the production by casting of components previously considered unsuitable.



**Halves of two different dies cast in 3% chromium-molybdenum-vanadium steel for the production of aluminium stampings.**

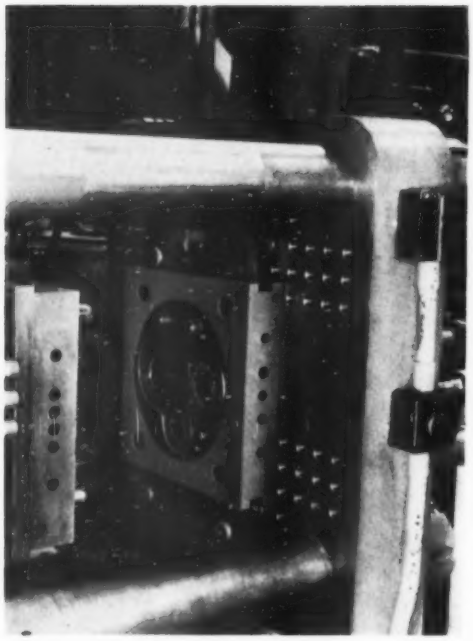
### Advantages of the Process

To appreciate the many advantages of the P.A.C. precision casting process, it is necessary to forget the limitations of structure and composition that for many years have doggedly pursued " casting " in its broadest sense. Later in this article, evidence is given regarding the structural properties and soundness of these castings. To this, may be added the fact that almost any type of steel may be cast by the process, and that practically any

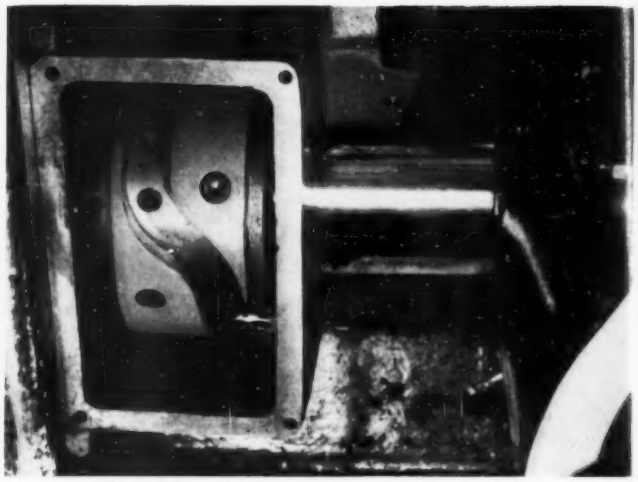
**A height micrometer, the anvil of which is cast in 1% carbon, 1½% chromium steel**

**Finished 6 × 20 in. scalping cutter.**

**Moulding die for plastic electric meter body cast in 3% chromium -molybdenum-vanadium steel.**



**Photograph taken at the Birmingham Small Arms Company's Small Heath Factory, showing a P.A.C. cast die on a PECO die casting machine**



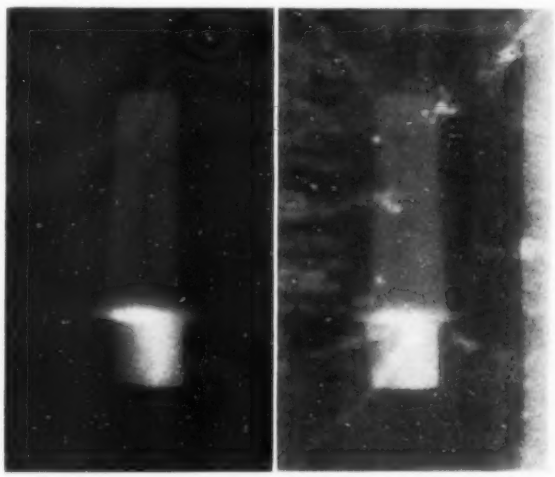
**A close-up of a B.S.A. Acme-Gridley multi-spindle automatic bar machine, showing the stock stop cam drum, which is fitted with P.A.C. cams cast in 1% chromium, 0·4% carbon steel.**

combination of mechanical properties can be obtained. With this in mind, it is easy to assess the value of the process from a purely practical point of view. Primarily, it enables any tool, die or component to be cast very close to its final shape, and even for components of high precision it is necessary to leave only a small amount of material for finish machining. It will be appreciated that a considerable saving in the total cost of production can be achieved, since there is immediately a reduction in both machining time and the use of machine tools. Rough machining is eliminated altogether, and the plant which is normally engaged on this operation can be re-allocated to finish machining—virtually doubling the capacity of the toolroom. It also means, of course, that the skilled toolmaker can concentrate on the most important part of his job, namely, that of finishing the tool or die. For some components, such as certain types of plastic moulding dies, the forms may be cast with a minimum of material left on, which may be removed by bench finishing or polishing. An additional saving lies in the cost of raw material, particularly when, as with most orthodox methods, a lot of metal has to be removed which is normally wasted.

A further advantage of the process is that P.A.C. castings have been found to give an equivalent, or even better, life when compared with items produced by more conventional methods. This has been particularly noticeable with extrusion dies for aluminium, brass, copper and steel, and also with stamping dies, where a life at least as good as that obtained from dies made by conventional methods has been obtained when producing stampings in a 13% chromium steel to S62.

Since castings can be produced from comparatively low-priced patterns, the process is equally economical for either large or small numbers of components, and very little capital outlay is involved. There is almost no limitation on the type of steel that can be cast ; in fact about forty different types of alloy steels are being used at the moment. These include case-hardening, constructional, carbon and alloy tool steel, and heat- and creep-resisting types, and it is possible to obtain almost any mechanical properties required by the relevant B.S.I. and D.T.D. specifications. Furthermore, special combinations of mechanical properties can be achieved, and, when required, a customer's own specification can be met. Conventional heat treatment can be applied to products cast by this process, and components or tools can be supplied in either the heat treated or fully softened condition.

To complete the list of advantages of this process, a word regarding accuracy is necessary. In this respect a limit of approximately ± 2% can be maintained on



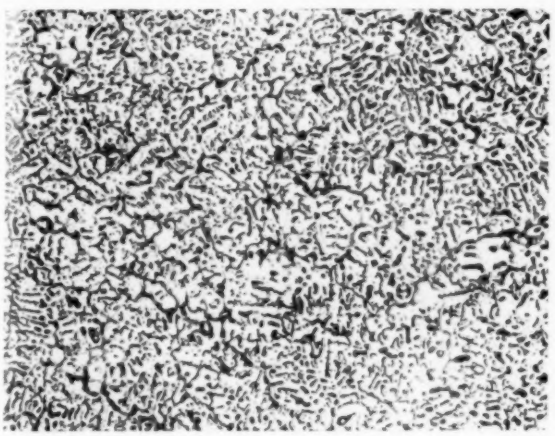
**Radiograph of cast stamping dies in 10% tungsten hot work steel. That on the left represents normal production, but porosity and internal cavities have been deliberately introduced into that on the right.**

important dimensions as a general rule, but this figure depends on both size and complexity. In certain cases it may be possible to improve on these figures.

### Soundness and Properties

Before detailing the precise structural properties of P.A.C. castings themselves, the results obtained on components produced by other methods, in particular, forgings and stampings, may be considered. Both forgings and stampings show "flow lines" in their structure which are often referred to as "grain". Whilst the mechanical properties along the flow lines are generally superior to those obtained in a casting, transverse properties may be inferior: this is particularly so with high strength materials. The properties of a casting are the same in all directions, so it is fair to say that, in general, castings give better "all-round" properties than a comparable forging or stamping.

For a number of applications forging is only employed as a means to an end, as in the production of plain blanks in alloy steel, which it would be uneconomical to produce by other conventional methods. In some uses of these forged blanks, it happens that the maximum



**Photomicrograph showing the structure of a P.A.C. casting. ×100**

working stress is applied across the grain, thus necessitating the use of a higher class material than is really necessary in order to obtain the requisite properties. Since the properties in a casting are non-directional, a lower class material can be used with an obvious saving in material cost.

The surface finish of the castings is excellent and the structure of the material shows a good, even, small grain size. For most commercial applications with open tolerances, the surface finish is sufficiently good to obviate finish machining operations. Fine tolerances, of course, necessitate some finish machining, but this can be reduced to a minimum with good design. To the customer, a further guarantee of soundness and quality lies in the facilities existing in the P.A.C. inspection department, which is able to carry out mechanical testing, and supersonic and radiographic examination, as well as closely checking dimensional tolerances and surface finish.

Whatever method is used to manufacture a component, the designer's main concern is that the component shall have the ability to stand up to the stresses and strains to

**Group of cast engineering components, some of which are used on B.S.A. machine tools.**

which it will be subjected during its working life. Due to the inherent nature of steel, the mechanical properties depend on the size of the component, but a whole range of steels with increasing alloy contents are used so that uniform mechanical properties may be obtained throughout a component by the choice of the correct steel. Castings can be supplied in the heat-treated condition if required.

TYPICAL COMPOSITION OF CAST STEELS.

| Series | Number | C% | Mn% | Cr% | Ni% | Ti% | W% | Mo% | V% |
|---|---|---|---|---|---|---|---|---|---|
| Case-Hardening Steels | 1 | 0·15 | 1·25 | 0·6 | | | | | |
| | 2 | 0·15 | 1·25 | 3·0 | | | | 0·3 | 0·15 |
| Constructional Steels | 1 | 0·3 | 1·25 | 0·65 | | | | 0·3 | |
| | 2 | 0·3 | 1·25 | 3·0 | | | | 0·3 | 0·15 |
| | 3 | 0·3 | 0·6 | 3·0 | | | | 0·5 | 0·15* |
| | 4 | 0·4 | 1·25 | 0·6 | | | | 0·3 | 0·15 |
| | 5 | 0·4 | 1·25 | 3·0 | | | | 0·3 | 0·15 |
| | 6 | 0·5 | 1·25 | 0·6 | | | | | |
| | 7 | 0·5 | 0·6 | 3·0 | | | | 0·3 | 0·15* |
| | 8 | 0·6 | 0·6 | 0·6 | | | | 0·3 | * |
| | 9 | 0·7 | 0·6 | 0·6 | | | | 0·3 | 0·15* |
| Stainless Steels | 1 | 0·2 | 0·6 | 13·0 | | | | | 0·25 |
| | 2 | 0·3 | 0·6 | 13·0 | | | | | 0·25 |
| | 3 | 0·4 | 0·6 | 13·0 | | | | | 0·25 |
| | 4 | 0·15 | 0·6 | 18·0 | 8·0 | 0·8 | | 0·4 | 1·0 |
| Carbon Tool Steels | 1 | 1·0 | 0·7 | 1·5 | | | | | |
| | 2 | 1·0 | 2·0 | 0·6 | | | | | 0·15 |
| | 3 | 1·25 | 0·7 | 1·0 | | | 2·0 | | 0·15 |
| | 4 | 2·25 | 0·7 | 13·0 | | | | | 0·15 |
| | 5 | 0·7 | 0·7 | 0·3 | | | 1·5 | | 0·15 |
| | 6 | 1·0 | 0·7 | | | | | | 0·15 |
| | 7 | 2·7 | 0·7 | 24 | | | | | 0·15 |
| High Speed Steels | 1 | 1·2 | 0·6 | 13·0 | | | 4·0 | | 1·0 |
| | 2 | 1·5 | 0·6 | 13·0 | | | 4·0 | | 2·0 |
| | 3 | 2·0 | 0·6 | 13·0 | | | 4·0 | | 4·0 |
| | 4 | 0·8 | 0·6 | 4·0 | | | 18·0 | | 1·0 |
| Nitriding Steels | 1 | 0·15 | 1·25 | 3·0 | | | | 0·4 | 0·2 |
| | 2 | 0·3 | 1·25 | 3·0 | | | | 0·4 | 0·2 |
| | 3 | 0·45 | 1·25 | 3·0 | | | | 0·4 | 0·2 |
| Forging Die Steels | 1 | 0·5 | 0·7 | 0·65 | 1·5 | | | 0·3 | |
| | 2 | 0·5 | 0·7 | 0·9 | 2·0 | | | 0·8 | 0·15 |
| | 3 | 0·5 | 0·7 | 3·0 | | | | 0·3 | 0·15 |
| Hot Die Steels | 1 | 0·3 | 0·6 | 3·5 | | Si% | 10·0 | 0·3 | 0·2 |
| | 2 | 0·35 | 0·6 | 5·0 | | 1·0 | 5·0 | 1·0 | |
| | 3 | 0·35 | 0·6 | 5·0 | | 1·0 | 1·3 | 1·5 | 0·2 |
| | 4 | 0·35 | 0·6 | 5·0 | | 1·0 | | | 1·5 |
| Heat- and Creep-Resisting Steels | 1 | 0·4 | 0·6 | 13 | 13 | 1·25 | 2·5 | | |
| | 2 | 0·15 | 0·6 | 23 | 14 | 1·25 | | 1·0 | |
| | 3 | 0·15 | 0·6 | 20 | 1 | | | | |

* Controlled sulphur and phosphorus.

### Examples of Applications

The field of application for alloy steel castings is constantly expanding and, as fresh applications are found, its scope will be extended still further. Already these castings are being extensively used in the jig, tool and die industry; extrusion dies, hot stamping dies, plastic moulding dies and pressure die casting dies have been produced very satisfactorily, which, in production, are achieving remarkable economy. In the general engineering industry, for such items as tools, tool-holders, impellor blades, machine tool parts, and many small and intricate components, the process is also being used to advantage. A selection of typical applications is shown in the accompanying illustrations.

### Design

The precision alloy casting process undoubtedly presents vast possibilities, and is yet only on the fringe of an extensive field of application. In the foundry, technical advances will continue to broaden the scope of the process, but one factor above all others will determine the extent to which it can be put to practical use, namely the degree of understanding and collaboration that is established between the designer and the founder. It is essential for the designer to appreciate in some measure the technicalities of the process and the special difficulties that face the founder. Most of these difficulties can be overcome quite simply by the right approach to the design of the product. By allowing the founder as much freedom as possible in modifications to the design (where these are necessary), and by co-operating fully with him, the designer will be assured of a first class product and will contribute greatly to the overall development of the process. To sum up, it is important that each job be treated on its own merits. The advice of the firm's technical staff, together with development facilities, are readily available to assist anyone wishing to investigate the use of precision alloy steel castings. Whilst strides are being made in the casting of steel, other possibilities are also being investigated, such as the casting of non-ferrous alloys based on copper and aluminium. Increased efforts are also being directed to the aircraft industry, where a successful application of precision alloy castings has already shown that many outstanding problems experienced in the specialised machining of high precision components can be overcome by the process.

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# Permanent Magnet Association Lecture

THE Third Annual Celebrity Lecture of the Permanent Magnet Association was delivered by Professor L. F. Bates, F.R.S., at the Royal Victoria Station Hotel, Sheffield, on Thursday, April 14th. The lecture was of particular interest to Sheffield metallurgists and the permanent magnet industry because it dealt with the development of the domain concept of ferromagnetism, initially postulated by Pierre Weiss to explain the fact that by the application of a weak magnetic field a piece of unmagnetised iron may easily be magnetised strongly. The iron is supposed to consist of a collection of small regions or domains, each strongly magnetised, but the directions of magnetisation of the several domains are so randomly orientated when the iron is in the unmagnetised state that they neutralise one another, while in the strongly magnetised state their directions of magnetisation are practically parallel to one another. In recent years, the domain concept has been considerably developed to account for some of the magnetic properties of single crystals of the ferromagnetic metals, of polycrystalline, rolled silicon-iron and, finally, of permanent magnet materials such as Alnico and Alcomax.

The most spectacular method of showing the existence of domains is the powder pattern method, a modern extension of the well-known method of plotting the field around a bar magnet by means of iron filings. On a highly polished and carefully annealed surface of a ferromagnetic crystal, a drop of a solution of colloidal magnetite is placed (or a very fine ferromagnetic powder is blown) when the magnetic particles collect above those regions of the surface where a boundary between two neighbouring domains intersects it. This deposition occurs because such boundaries separate domains whose directions of magnetisation differ; e.g., in the case of iron the angle between two such directions is usually 90° and 180°, and so we talk of 90° and 180° boundaries. Because the direction of magnetisation within a boundary changes continuously in passing from one side of the boundary to the other, a stray field must exist wherever a boundary cuts a surface.

The powder pattern technique allows us to show that a single crystal of a ferromagnetic metal may be pictured as made up of the main domains just described, together with a number of small, subsidiary or closure domains, whose function is to avoid unnecessary surface magnetisation of the material. Many lovely photographs of the patterns formed by the walls of such principal and closure domains have been obtained, and the study of these photographs has proved conclusively the essential correctness of contemporary views on magnetisation processes, and has also enabled us to make new fundamental experiments in magnetism. Professor Bates and his co-workers have made very many investigations of this kind and have paid much attention to numerical calculations based on such work, for which painstaking and accurate preparation of the metal surfaces is imperative.

It is extremely interesting to see a film which illustrates the experimental technique, and to watch the formation of domain patterns in a changing magnetic field, especially upon the surface of a crystal of cobalt, as in this material the domain structure is very pronounced, as well as being the simplest known. It is also striking to view photographs which show how the motion of domain walls is impeded by the presence of impurities or defects in a material, and to discuss the importance of these phenomena re the performance of transformer materials.

Of special interest to a Sheffield audience were some pictures taken of domain patterns on the surfaces of specimens of Alnico and Alcomax. Such pictures have not hitherto been obtained with these materials in the treated state, when they are suitable for permanent magnets, but Professor Bates and Mr. P. F. Davis have very recently obtained valuable photographs which, for example, show conclusively the difference in structure between a crystallite of Columax in the untreated state and during the different stages of the treatment required for the optimum permanent magnet properties.

# Control of Quality in Heat Treatment and Final Operations in the Production of Wrought Non-Ferrous Metals and Alloys

## Report of the Third in a Series of Institute of Metals Symposia

THE third of a series of whole-day symposia on the Control of Quality in the Production of Wrought Non-Ferrous Metals and Alloys was held at Church ouse, Westminster on Thursday, March 31st, as part of e Spring Meeting of the Institute of Metals. The airman at the morning session was Dr. Maurice Cook, resident of the Institute.

The discussion was based on the following papers, ublished in the February, 1955, issue of the *Journal of e Institute of Metals*.

*Assessment of Quality in Wrought Products*, by DR. W. G. SHILLING (Aeronautical Inspection Directorate).

*The Control of Quality in Heat Treatment and Final Operations in the Production of Rolled, Extruded and Drawn Aluminium and Aluminium Alloys*, by MR. A. J. FIELD and MR. J. SALTER (British Aluminium Co., Ltd.).

*Heat Treatment and Finishing Operations in the Production of Copper and Aluminium Rod and Wire*, by MR. H. J. MILLER (British Insulated Callender's Cable Co., Ltd.).

*The Control of Quality in the Heat Treatment and Finishing of Copper and Copper-Base Alloys*, by MR. V. B. HYSEL and MR. T. W. COLLIER (Thomas Bolton & Sons, Ltd.).

*The Production of Light-Alloy Drop Forgings, their Heat Treatment, Inspection and Testing*, by MR. W. T. EDMUNDS and MR. R. C. LLOYD. (Light Metal Forgings, Ltd.)

*The Heat Treatment, Inspection and Testing of Wrought Nickel and Nickel Alloys*, by DR. W. BETTERIDGE (The Mond Nickel Co., Ltd.) and MR. T. E. COUND (Henry Wiggin & Co., Ltd.).

As the above papers are already available in printed form, no further details will be given in this report, with the exception of references made by the rapporteur in his opening remarks and by subsequent speakers in the discussion.

### MORNING SESSION

#### Rapporteur's Introduction

DR. A. R. E. SINGER (Birmingham University), who acted as rapporteur, referred to the fact that the symposium was the third in a series dealing with quality control in the production of wrought non-ferrous metals and alloys. The first dealt with melting and casting and the second with working operations, so that the present symposium was the logical conclusion of the series.

In the main, the contents of each of the seventeen papers presented in the series could be divided into two main aspects. The first concerned the essential "know how" of the manufacture of non-ferrous metals, and the second the institution of measures for controlling quality. On the manufacturing side, an enormous range of ideas on equipment and techniques necessary to produce acceptable products had been given, and DR. SINGER felt that the generous attitude of the companies associated with the papers should be recognised, in that they had allowed this information to be given without reserve, and had agreed to its being made available to the world through the medium of the *Journal of the Institute of Metals*.

The information was essentially detailed, which meant that it was necessarily of a certain localised interest. By the very nature of things, the people dealing principally with nickel and nickel-base alloys used techniques which were not wholly applicable in other fields, but the interchange of information helped in the spreading of ideas between the various groups of workers.

DR. SINGER thought that it had been generally recognised in the papers in the past that considerable economic benefits could often be obtained by using to the full some system of quality control, but in the present series of papers there were fewer examples of control of that kind. He believed that finishing operations were not unsuitable for control methods, but thought their full potentialities in this field had not been realised.

Turning to the individual papers, the first, by DR. SHILLING, on the "Assessment of Quality of Wrought Products" dealt principally with aeronautical inspection. The author outlined the administrative procedure and the methods of working adopted by the A.I.D., giving a great deal of information on the utilisation of technical methods, mentioning in particular those for chemical analysis, tensile testing and non-destructive testing.

DR. SINGER said he was particularly interested in the following three sentences from the middle of the paper: "Tests called for in the specification do not always yield results that are of direct use to the designer. It must be remembered, however, that the aim of the specification is to provide uniform material. The designer's requirements can be catered for elsewhere." There was a tendency to lose sight of the fact that a specification aimed at producing uniform material, as many specifications were couched in terms of minimum requirements. They really formed one aspect of control.

The next paper, by MESSRS FIELD and SALTER, was a very comprehensive one dealing with "The Control of Quality in Heat Treatment and Final Operations in the Production of Rolled, Extruded and Drawn Aluminium and Aluminium Alloys." It concerned all aspects, including flattening, shearing, annealing, heat-treating and packing, and of particular interest was the grain size assessment they used, based upon the Erichsen test, for the long-term control of grain size. Also of particular interest was reference to partial annealing or temper annealing or letting down, which, given suitable control measures, could lead to big economies in the production of intermediate hardness material.

The papers by MR. MILLER and by MESSRS. HYSEL and COLLIER discussed the annealing and heat treatment of copper and copper alloys on an industrial scale, and paid special attention to problems of bright annealing and, also, in the case of the paper by MESSRS. HYSEL and COLLIER, to stress relieving by annealing.

The paper by MESSRS. EDMUNDS and LLOYD differed from the others in that it dealt with light alloy forgings.

A very interesting section of the paper concerned the choice of forging stock relative to the size of and the properties required in the final product. On the heat-treatment side, some valuable data were given on the avoidance of internal stress and distortion by quenching the forgings from the solution treatment temperature in water held at various temperatures. Information was also given on the effect of such treatment on the mechanical properties. The final section of the paper dealt with defects which may occur in forgings and their methods of detection.

In the paper by Dr. Betteridge and Mr. Cound, dealing with nickel and nickel-base alloys, the metallurgy was quite different and the control methods employed unconventional. Considerable use was made of interstage examination during manufacture, and of a number of special testing methods and novel simulated tests. Some of the unorthodox ideas might well find application in other fields.

In conclusion, Dr. Singer said he would like to see some thought given to the institution of more control processes in the final stages of manufacture. It was not easy to do, but that was no good reason for not doing it, and he felt that any progress made would be of lasting benefit in the field of non-ferrous metallurgy.

## Discussion

Mr. S. S. Smith (Research Manager, Imperial Chemical Industries, Ltd., Metals Division), in opening the discussion, referred to the first paper presented to the three-part symposium in 1953, when Dr. Singer dealt with the principles and application of statistical quality control. This aspect seemed to have received little attention, as only two subsequent papers (none in the present series) had provided serious mention of the subject. It might have little significance in dealing with ingots, and when the count was small, but in finishing operations, where the count was inevitably higher, circumstances were different, and the consumer might be better satisfied if statistical quality control were used as a guide to maintaining uniformity of product.

The distinction drawn by Dr. Singer between control and activities concerned with investigating an unstable process, and with the making of appropriate modifications to a process, had been put in another way by Mr. Kasz last year when he thanked authors for advancing knowledge of what we do, but wished that greater emphasis had been laid on the control of what we do. Mr. Smith thought that Mr. Kayser was right when, on a previous occasion, he argued that statistical methods must be used by the man in the works, for without such control two inspection departments were necessary, one at the producer end and the other at the consumer end. To cut down such duplication, there was need for a free exchange of views between the two parties to the transaction, and it was evident that effective control, of whatever type, should be aimed at producing material appropriate to end use, and capable of being turned out at an economic price.

Dr. Shilling, in an admirably objective fashion, had defined the requirements under a number of broad headings. Referring to one aspect, control of chemical composition, Mr. Smith said a brief reference was made to the subject in some of the papers, but there was little comment on the standards and accuracy of the control procedures involved, or on the specifications, as such, with which the writers were concerned.

In commenting on individual papers, Mr. Smith said that he had selected two features, namely surface finish and the tools used in achieving that finish, for special attention. Messrs. Field and Salter had touched upon details which were often neglected in describing how finish was maintained, and to a certain extent it appeared that responsibility for control of quality resolved itself into controlling the men doing the job. He would like to ask the authors two questions: firstly, "Had they made use of devices such as those suggested by the R.A.E. for the assessment of flatness in sheet?"; and, secondly, "Had they any views on the use of controlled atmospheres for minimising staining of aluminium-magnesium alloys during annealing, or did they prefer flash annealing?" The subject of staining in heat-treatment was still of vital importance.

At one point Mr. Miller mentioned rough annealing and quenching as a means of improving surface, but Mr. Smith wondered whether he and other authors paid due attention to their choice of tool materials, with a view to producing a fine finish. This was a vital matter and one which the Institute and the B.N.F.M.R.A. were looking into in a serious fashion. Two questions directed to Mr. Miller concerned the use of die scalping tools in the making of co-axial tape, and the effect of drawing die shape on the extent of spring back.

In the interesting paper by Messrs. Hysel and Collier, the authors had chosen to emphasise special materials rather than copper and brass, which represent the major output of the industry. Their statements were authoritative, although there was a tendency to oversimplify. There was, for instance, a reference to the fact that in the production of 1·31 in. wide copper strip a lateral contraction of 0·0004 took place, and that allowance had to be made when preparing the cutters for slitting hard strip before annealing. This certainly indicated that attention of a high order was paid to tools. In view of the prime cost of the materials involved in the nickel alloy field, ruthless rejection of faulty material at the earliest possible stage was necessary, and Mr. Smith was particularly interested in the specialised techniques used for control.

Reverting to tools and tool materials, Messrs. Edmunds and Lloyd gave few details of the die steels used. The tools must be very costly, and he felt that a large number of products coming from the hammer offered scope for statistical quality control. "Was this in fact applied?"

In conclusion, Mr. Smith was pleased to note the attention paid by all authors to packaging as the final gesture to win the confidence of the customer.

Dr. E. A. Bloch (Aluminium Industrie, A.G., Neuhausen, Switzerland), said that the high standards of quality in Britain, even by Swiss standards, made it difficult to add anything new to what had been said. He, therefore, limited his remarks to the subject of non-destructive testing, with emphasis on ultrasonic techniques. Their development of this method arose as a result of difficulties in fabricating certain extrusions in S75S alloy for the Swiss aircraft industry. Application of the technique to the bigger sections and simpler shapes in the as-cast condition enabled defective material to be rejected before expensive work was carried out on it. Using an instrument working on the reflection principle, it was possible to determine cracks, inclusions, gross segregation, porosity, etc., but the method was perhaps too sensitive, in that micro-porosity, which does not affect the quality

of the extruded product, was also found. Information from the United States described an automatic precision scanning instrument which could use higher frequency waves and detect faults at a depth of one-eighth of an inch from the surface.

In conclusion, DR. BLOCH asked for an explanation of the two limits of proportionality in the stress-strain diagram for aluminium-clad aluminium alloys, referred to by DR. SHILLING, and for further particulars of an electronic instrument for testing tubes, described by MESSRS. FIELD and SALTER.

MR. E. J. BRADBURY (Development Metallurgist, The Mond Nickel Co., Ltd., Development and Research Department), in commenting on the implication contained in DR. SHILLING's paper that inspection must be carried back to the very beginning of any processing, suggested that in doing so the inspection authority must lay down the grade of processing that must be carried out, and he questioned the ethics of the authority then rejecting material which had been processed in accordance with its instructions.

As many of the most important elements added to or eliminated from materials were not covered by specifications, he wondered whether the A.I.D. were not deceiving themselves in reading the analysis figures supplied, and whether they should not rather just accept the certificate that the material was what it purported to be. Whilst on the question of composition, MR. BRADBURY referred to the problems arising as a result of the production of materials in very small batches by powder metallurgy methods. MR. MILLER had mentioned that strand annealing copper was slow, presumably because it could not be speeded up by the use of hydrogen, and he wondered whether the possibility of eliminating defects such as sticking or discolouration by the use of vacuum annealing had been examined. MR. BRADBURY repeated MR. SMITH's request to MESSRS. EDMUNDS and LLOYD for data concerning die life, particularly in its relation to the shape or the deformation that the material received, whether it was related to the mass of the forging being handled, or was solely related to the temperature which had to be used.

In referring to the paper by DR. SHILLING, DR. G. E. A. BRAMLEY (Chief Metallurgist, Aluminium Wire and Cable Co., Ltd.), urged the adoption by the A.I.D. of the nomenclature used by the British Standards Institution and the industry to designate the condition of the material. The principle mentioned by MESSRS. FIELD and SALTER of giving reasonably heavy reductions in cold working to produce a fine grain size after heat treatment applied in the drawing of aluminium alloy wire, but there were limitations in the case of certain alloys. With multi-die continuous drawing machines, it was desirable, in order to secure their maximum utilisation, to impose overall reductions in area of upwards of 80% from inlet to outlet size. With some alloys, however, reduction beyond about 70% resulted in isolated large grains after solution treatment, such irregularities being unacceptable in applications where cold forming operations were involved, but with multi-die machines it was more economical to draw with maximum reduction and then let-down to the intermediate temper than to draw to an inter-anneal size and follow by a temper drawing operation. The increased ductility of temper-let-down material was also advantageous and the reduced handling minimised the possibility of process damage.

The temper-letting-down of electrical purity wire for certain types of insulated cables, mentioned by MR. MILLER, was somewhat more complicated, since it was desirable to heat treat the wire on the steel stranding reels on which it was normally drawn direct, and the heat capacity of the steel reel could affect the uniformity of the partial anneal achieved. The problem of burnt oil stain after annealing was a serious one, particularly with thicker gauge wires, where relatively high viscosity drawing lubricants were used. Burnt oil stain was particularly harmful on wire to be flattened to strip, as it affected the friction in the roll gap and influenced the degree of spread. Strand annealing might be of assistance in this problem, and it would be interesting to hear any experiences with the method, mentioned by MR. MILLER for copper wire, of strand annealing in tandem with wire drawing. Difficulties in producing wire in natural ageing alloys with a fairly high degree of uniformity of mechanical properties, after a limited period of natural ageing, were found to be due to water entrapped in the coil after quenching, ageing being retarded until all the water had evaporated.

In the production of high-strength heat-treatable aluminium alloy wire in straight lengths for machining on automatics, the material was solution treated in coil prior to straightening, and difficulties were sometimes experienced, due to the coil settling down by its own weight at the solution treatment temperature, resulting in the formation of short kinks, or even indentations, where one lap crossed another. Such kinks could not be removed in the usual type of spinner straightening machine.

He could not agree with MR. MILLER's general statement that extrusion resulted in a superior surface condition of the drawn wire as compared with that produced from hot rolled rod. In fact, he would say that wire with the highest quality of surface finish could be produced from hot rolled material. With the growing use of aluminium for electrical purposes, increasing quantities were now being drawn in works which had previously handled only copper, and DR. BRAMLEY concluded by stressing the desirability that the two materials should be kept separate, as trouble had been caused by copper dust settling on aluminium wire.

MR. J. F. KAYSER (Chief Technician, Gillette Industries, Ltd.), said that the fact that he was only interested in mass production, where the true end product was dollars, would influence everything he had to say. From that point of view, if he wished to produce a really good cheap saucepan from aluminium, the sort of rubbish that went into an aeroplane would be no good at all. It would not be sufficiently uniform. In discussing the role of the inspection department, MR. KAYSER said it was the duty of production departments to pass along satisfactory material, so that final inspection would merely check, and rejection should be the exception rather than the rule. To this end it was essential that the production department should observe what it was producing and make certain that the product was under control. It was, therefore, necessary to make measurements the whole of the time.

On the question of uniformity of product, MR. W. W. KEE (Head of the Laboratories, Enfield Rolling Mills, Ltd.), said it was not only a question of uniform material within a batch, or from one batch to another: what the customer wanted was to be able to buy material next

June of the same quality as that bought today. Discussing the question of edgewise rolling, MR. KEE asked how MESSRS. FIELD and SALTER took a sample for measuring the straightness, and what extra cost was involved in changing from 1 in. on 10 ft. to ¼ in. on 10 ft. He was also interested to know how many standards of surface quality the same authors had, how they were measured, and for what end uses the division was made.

With a critical amount of cold working, and with uneven cold working, excessive grain growth could be obtained in chromium copper, and he wondered to what extent these factors influenced the growth referred to by MESSRS. HYSEL and COLLIER. He was rather surprised at the precipitation-hardening treatment, quoted as 475° for 4 hours, as, in the absence of certain impurities or other elements, he would have expected appreciable over-ageing.

MR. H. CHADWICK (Metallurgist, Northern Aluminium Co., Ltd.), confined his remarks to one aspect of the paper by MESSRS. EDMUNDS and LLOYD, namely that dealing with the occurrence of blisters on forgings manufactured from extruded stock, particularly in L64 or L65. It was difficult to decide the cause of the blisters from their appearance on the surface of the forging, but he believed that they could be divided into four main types, microscopic examination affording a ready means of differentiation. In those due to over-heating, the characteristic rounding of the grains caused by partial melting could readily be seen, and etching in 25% nitric acid showed the degree of overheating by the width of the light bands crossing the dark background. Other causes of blisters were insufficient lubrication in the die during forging, faulty stock, and reaction with the furnace gases during solution heat treatment. The last two types showed no evidence of over-heating. There was some evidence that in the production of blisters during forging, heat was developed at the moment of impact along the planes of maximum deformation, blisters developing during subsequent solution heat treatment. It was possible to reduce blistering to a very low level by one or other of the following measures: adequate temperature control during preheating and solution heat treatment; the rejection or scalping of defective stock; the use of creosote as inhibiting agent during solution heat treatment; and effective lubrication during forging.

MR. A. R. MARTIN (Chief Metallurgist, Northern Aluminium Co., Ltd., Birmingham), pointed out that there was still a strong element of art involved in forging, particularly in the preparation of the dummy or use. As mentioned by MESSRS. EDMUNDS and LLOYD, pre-forming dies were the best, and, in many cases, not necessarily the most expensive answer. There was much to be said for interstage removal of defects, as those carried over from one stage to the next became progressively more difficult to detect and remove. Furthermore, steps could be taken to modify the technique on recognition of systematic types.

No reference had been made to polishing followed by chromic acid anodising as an inspection technique which was extremely sensitive. Polishing was time-consuming and expensive, however, and it would be very useful if the eddy current method of surface flaw detection could be developed for forgings, although this raised problems of probe design for contoured surfaces. The cast billet stage had been stated to be the best for carrying out ultrasonic flaw detection, and whilst it was true that for major oxide inclusions and cracks, it was possible that very small spherical defects would be more easily found after spreading during subsequent forging. Probe design was again a problem, the single probe technique being the easiest to use on irregularly shaped forgings: for detecting flash line defects in larger forgings, the sheer wave probe, designed for seam welds in large diameter pipes, had proved very successful.

The long list of defects which may be associated with extrusion would suggest that such material was likely to be the least satisfactory starting point for forging manufacture. Probably the majority of small and medium size forgings were produced from such material, however, and where longitudinal properties were of the greatest importance, and the shape of the forging lent itself to production from extruded material, it was, of course, superior. The longitudinal properties of rolled bar were not inferior to those of extruded bar, but there was no significant difference in forgings made from the two materials. The best combination of properties was undoubtedly achieved by triaxial working of cast material on a press, when improved transverse properties, particularly elongation, were obtained in the forgings.

Non-destructive testing was again discussed by MR. K. E. THOMPSON (Metallurgist, Northern Aluminium Co., Ltd., Banbury), who drew attention to a number of disadvantages of the ultrasonic method: (1) it was not always possible to distinguish between a cluster of small holes strategically placed and a major defect; (2) certain undesirable discontinuities did not necessarily constitute an ultrasonic discontinuity; (3) it was difficult to measure the size of a defect ultrasonically with no knowledge of its orientation or configuration; (4) the methods did not really lend themselves to the examination of complicated extruded shapes; (5) it was sometimes difficult to maintain a uniform volume of oil under the probe, and a uniform coupling was essential in order to obtain consistent results. The last objection could be overcome by adoption of the immersion method mentioned by DR. BLOCH, which also facilitated automatic movement of the probe. The method was still, however, comparatively slow and laborious, and industry was seeking quicker and more precise non-destructive tests. The eddy current method could be regarded as complementary to ultrasonics, and MR. THOMPSON sought information as to whether the method was restricted to the examination of round shapes, and what difficulties might be expected with other regular sections such as squares and rectangles. Despite the present limitations there was no doubt of the value of ultrasonic testing, and although eddy current testing was very much restricted in its application, the two methods together would form a very important part of the inspection programme in the future.

MR. A. W. MATTHEWSON (Chairman of the Chief Inspectors Committee and Chief Inspector of Birmetals, Ltd.), commenting on the paper by MESSRS. FIELD and SALTER, said he did not believe one should have all the inspectors within one organisation. In his firm there was one inspection based on the A.I.D. inspectorate, and over and above that a statistical quality control of the processing which was presented at each weekly production meeting, so that trends could readily be seen. On the inspection side, even with aircraft materials made according to specification, a regular exchange of views

with the customer was essential, because from customer to customer there were differences in requirements. On the commercial side, many customers ordered a material for a certain product, and it was no use saying it was to specification if it did not do the job. MR. MATTHEWSON concluded by emphasising that inspectors should be completely independent of production, and that their job was not to reject material but to make certain that material going to the customer was usable and suitable for use in the end product concerned.

MR. T. W. BUSHELL (Principal Surveyor for Metals, Lloyds Register of Shipping), stressed the importance of the inspection staff being independent of the production staff, and pointed out that the question to be answered in all inspection was "Will this part perform the service for which it is intended?" Reference had been made to certain alloys as being "treacherous," and there certainly appeared to be individuals in certain groups of metals which might be described as unreliable. Certain idiosyncrasies were present, many of which were not always appreciated by the designer. For such reasons the control of quality was even more important than was sometimes appreciated. There was no doubt, too, that independent inspection organisations would find satisfaction in realising to what extent control of the manufacturing processes and the various items of works inspection had developed.

There were one or two points on which he would be glad to have further information. It would be interesting to know, for instance, what effects the stretcher levelling process had on the physical properties of the material so treated, and whether, as in the case of ferrous metals, gradual recovery took place. The narrow temperature ranges used in the heat treatment of light alloys raised questions of temperature control, particularly in view of the fact that at the low temperatures involved it was not possible for an operator to see when the temperature was attained and when it was uniform.

In view of the fact that quality appeared to be the first consideration in this field, he was interested to note in the paper by MESSRS. FIELD and SALTER that one method of solution treatment was preferable to another on the score of economy, but was suitable only for medium-strength alloys. He assumed that such a change of procedure would not be instituted merely on the grounds of economy if an inferior product resulted. On this question of economy, MR. BUSHELL stressed that designers and users must recognise that where circumstances dictated the use of higher-quality products than those currently available, increased first cost must be faced, but there were many cases where the dearest was the cheapest in the long run.

Reference had been made to some difficult machining problems when dealing with extruded bars, and it would be interesting to know whether the distortion occurring during heat treatment also was greater in extruded than in rolled bars and sections. It was interesting to note that electronic testing had been instituted in the case of tubes made by the port hole type extrusion dies. In view of the fact that in certain circumstances the extrusion defect, even on solid sections, could extend for a considerable distance along the bars, had this method of testing been applied to such sections when they were intended for important structural members.

The paper by MESSRS. EDMUNDS and LLOYD emphasised the need for the utmost vigilance in all operations concerned with the manufacture of non-ferrous materials, and it would appear that the amount of time and money spent on the inspection side was not by any means a luxury but a necessity. The investigation of many failures indicated that in only a small minority could the primary cause be traced to the metal itself, and MR. BUSHELL felt that if the same thought and care were given to design and to methods of fabrication in the creation of machines and structures, as to the making of the metal itself, the number of failures encountered in practice might be reduced very considerably.

### Authors' Replies

Referring to DR. BLOCH'S remarks, DR. SHILLING said that a further point in favour of ultrasonic testing at the billet stage was that certain defects were not responsive to the technique after working, although in some cases the reverse was true. On the question of the two proof stress limits in clad material, DR. SHILLING said there were two yield points: the first for the cladding; and the second for the base material. He did not agree with MR. BRADBURY that if the A.I.D. inspected every part of a process they should accept the final result, whether it was good or not. They were not responsible for the process, and if it did not give the right answer the manufacturer would no doubt change it. Similarly, they were only really interested in the final chemical analysis. It was true, as MR. KAYSER had said, that some specifications were rather wide, but even though the content of a particular element allowed might vary between 3 and 6%, each manufacturer would set, much closer limits for his own working, such as, say 5·45 to 5·55%.

A few years ago non-destructive testing could be divided particularly into two classes, radiology and the rest, meaning that radiology was well established and that the rest were unknown. Today ultrasonics could be placed midway, and with a little more experience and knowledge it would join radiology. Other techniques using eddy currents, high frequency bridges, etc., were progressing, but were still at the stage where, if nine out of a possible ten factors were absolutely invariable, the other one could be checked.

MR. EDMUNDS said that there was a difference between forging practice in the United States and in this country, where, owing to the reduced demand, the production of forgings was strictly individual. There was not, therefore, the same demand for intensive instruments for the detection of flaws, and in any case he felt that quality was expressed chiefly in the material supplied to the forger. He was in agreement with everything being done to ensure perfectly good forgings, including such methods as chromic acid anodising, but felt that it might be more economical to carry out such examinations at an early stage. Rolled bar was certainly good stock material, but it was their impression that nowhere in this country was it possible to obtain it. In reply to Mr. Smith and Mr. Bradbury, die life was a matter of design and mass; temperatures were secondary. If there were sharp lugs and bosses, they were bound to cause cracking of the die.

In the short time available, MR. SALTER said he would deal with one or two general points that had been raised. Reference had been made to his company's practice of having within its factories two inspection organisations. Their main inspection department, as distinct from that

dealing with aircraft materials, was not concerned in any way with production, but was responsible to the management, and its function was to see that standards not set necessarily by outside bodies, except through customer contact, were maintained. They felt that if they did not have an independent inspection department for the rather specialised materials supplied to the aircraft industry, and indeed some of the other defence services, those inspectors would become less specialised in their knowledge of the requirements of those particular industries. It would be confusing for the inspectors to have to switch their minds from one standard to another.

Many of the processes dealt with in this series of papers, such as guillotine shearing, roller levelling, stretching and so on, did not lend themselves to measurement of errors as the process proceeded and the taking of steps to correct an error, but they did, as effectively as possible, exercise statistical control of quality and of production conditions. The process of temper letting-down did not lend itself to automatic control, because it was so much affected by the prior thermal and cold working history of the material, which could vary throughout a one or two ton coil.

He was sorry if they had given the impression that the final operations with light alloy sheet and extruded sections were easy to control. They had got used to them, but constant vigilance was necessary. There were many differences in products for different uses, but for any particular specification their aim was uniformity. They had no experience with the application of the R.A.E. assessment of flatness, the company's method being to judge by viewing at a very acute angle to the sheet surface.

There was some difference of opinion among his colleagues as to the precise influence of controlled atmospheres on the surface produced, but he understood that such furnaces had been developed in North America specifically for aluminium-magnesium alloys, because with modern high speed strip rolling methods the strip was inevitably left with a substantial film of oil on it, to drive off which required a fairly long time at a fairly high temperature, conditions detrimental to the magnesium-containing alloys. It was thought by some of his colleagues that the controlled atmosphere in itself would reduce oil staining, as distinct from oxidation staining of magnesium alloys.

## AFTERNOON SESSION

The meeting continued in the afternoon under the chairmanship of Mr. W. J. Thomas, Chairman of the Metallurgical Engineering Committee of the Institute.

### Discussion

Mr. F. King (Chief Metallurgist, Northern Aluminium Co., Ltd.), in continuing the discussion, said that several of the authors had again made reference to the great importance of control in the early stages of fabrication, but it must not be forgotten that final operations were also responsible for a high percentage of the rejections experienced on final inspection. The process of temper annealing or temper letting-down provided an economic means for the production of intermediate tempers from materials primarily designed for strip rolling with heavy reductions. Temper annealed sheet had a bright finish, improved ductility and, provided prior conditions were suitably adjusted, improved formability. The disadvantage were oil staining and shape problems, and the greater care required in handling. Depending on thermal history and the amount of cold reduction, the temperatures employed for the treatment could be limited to a range of $\pm$ 15° C. down to $\pm$ 2½° C., in which case precautions taken with solution heat treatment loads would be equally applicable.

Where batch-type furnaces were found unsuitable, due to lack of temperature uniformity, air sheet heat treatment furnaces with well-spaced suspended loads had proved of value. Materials with wide temper annealing temperature ranges had been successfully flash annealed. Mr. King suggested that the defects mentioned by Mr. Miller in batch temper annealing wire might be overcome by strand annealing. Of the various methods employed for process annealing, the vacuum annealing process appeared to be the most intriguing, and further information would be welcomed. Problems such as joining coils for continuous annealing of aluminium and aluminium alloy strip were of a minor nature compared with those likely to be experienced with the handling of soft hot strip, and it would be of interest to hear how the copper and brass industry had dealt with such problems.

There was little doubt that there were advantages to be gained from the use of vertical heat-treatment furnaces for tubes, by way of improved quenching efficiency and reduced distortion. Tubes less than 1 in. diameter could be straightened by stretching, and larger sizes by a 5% drawing reduction. Mr. Miller had suggested salt bath furnaces as an alternative to batch furnaces for the solution heat treatment of wire, but serious corrosion would be encountered in the absence of efficient means of salt removal, and the oxide film formed during salt bath treatment could be a source of heavy die wear on subsequent manipulation.

He would agree that the solution of many oil staining problems lay in the development of improved techniques for oil removal prior to thermal treatment, and methods used for the control of lubricants was a subject coming within the scope of the present symposium.

Very little had been said in the papers on copper and nickel alloys on the control of quality in the pickling and cleaning operations, and some discussion on the subject would be welcomed. Turning to the importance of testing procedure, Mr. King endorsed Dr. Shilling's comments that tests called for in specifications did not always yield results of direct use to the designer, a fact not always fully appreciated by the latter. To avoid the necessity for an excessive amount of mechanical testing, the aim of quality control should be to enable the mechanical properties in any location to be predicted with reasonable certainty from the results of a limited number of tests.

In reading through the papers, the figures quoted for grain size standards seemed at first sight to be at variance with their own experience, but this might be accounted for by different methods of measurement. The same difficulty had been experienced with earing values, and these two subjects might receive further consideration, along with that of standards for flatness.

### Authors' Comments

The Chairman suggested it might be appropriate at this point to hear from one or two of the authors.

Mr. Miller said that both Mr. Smith and Dr. Bramley had raised the question of surface quality, in assessing which there was, unfortunately, no standard.

It had become obvious during the last few years that the copper and aluminium industries had decided on different techniques in producing good surfaces. The copper industry had decided that the best procedure was to break down the wire bar to $\frac{1}{4}$ in. or $\frac{5}{16}$ in. diameter in as small a number of passes as possible, thus producing a product good enough for more than 90% of copper wire requirements. Where high quality was required, a surface rectification process such as shaving had to be adopted. The aluminium industry had moved in the other direction, taking gentle passes to avoid turning in of fins.

On the question of spring back, raised by MR. SMITH, he would say that it was not influenced by the method of drawing or by the tools, because it was encountered only with annealed products. It was definitely dependent on conditions during annealing, and not damage introduced in the course of subsequent operations.

Vacuum annealing plants had been marketed by at least two Continental manufacturers, the greatest claim for them being the avoidance of stickiness of wire. The reason for its occurrence was not known but it might be due to the highly activated surface produced by reduction of the oxide film on the copper in the bright annealing atmosphere, so that pressure welding could occur. It was MR. MILLER'S impression that uneven spread in rolling wire into strip was entirely due to the very heavy mineral oils used for drawing aluminium wire, and the problem emphasised the need for further efforts to develop water-soluble lubricants.

DR. BETTERIDGE, in reply to MR. THOMPSON'S query on eddy current testing, said that they only used it for round bars, but it would be applicable to other sections, even though the sensitivity would vary at different points due to the variation in density of the eddy currents with the changing section. On pickling, MR. COUND, said that the essence of the process was completely to remove the oxide skin, so that the control of quality meant that all the skin must be removed. The picklants used in the works varied with the type of alloy, and methods used for nickel-chrome alloys and Nimonic alloys were mentioned in the paper. Formerly, for hot rolled sheet, immersion in alkaline potassium permanganate was followed by the so-called Monel pickle (sodium nitrate dissolved in sulphuric acid), but for cold rolled sheet with the oxide rolled into the alloy, an aqueous solution containing 5% hydrofluoric acid and 20% nitric acid was used, sometimes in conjunction with alkaline permanganate. More recently, immersion in molten caustic soda containing 10% sodium nitrate, followed by the nitric-hydrofluoric pickle had been introduced.

MR. HYSEL, said he was sorry that they had conveyed the impression that metal manufacture in any of its phases was a simple problem. Lack of space had prevented more details being given of the annealing of copper and brass strip. The question of accuracy in slitting had been referred to with the suggestion that 0·0004 in. was insignificant. With normal commercial tolerances on width, it would be, but in the particular case concerned statistical examination of the width of several batches of strip showed that a movement across the histogram scale of 0·0004 in. produced a greater percentage of acceptable material.

In reply to MR. KEE'S comments on chromium copper, MR. HYSEL said they had not experienced much trouble with abnormal grain growth in round and square section rod, but uneven amounts of cold work across a section might be a possible explanation in the case of commutator bars. They, too, had found silicon advantageous in preventing over-ageing, so that a temperature of 475° C. was not excessive.

On the question of pickling, MR. HYSEL said he would like to go a stage further back, to the control established during the last decade on the final annealing and the use of more suitable lubricants, both producing material less susceptible to the old trouble of the press manufacturer of fire staining. They had simplified the problem by producing satisfactory pickled strip. Developments such as continuous pickling, strand pickling, control of pickle solutions and the use of electrolytic recovery plants had all tended to produce a more uniform process.

MR. KING had mentioned the use of stretching to overcome subsequent distortion in machining. They had no experience of this technique, and the example cited in the paper referred to rather heavy blocks of phosphor bronze which were not really suitable for stretching.

### Further Discussion

MR. N. I. BOND-WILLIAMS (Aston Chain & Hook Co., Ltd.), said that, although it might appear a heresy to inspectors, he felt that the separation of an inspection function into a water-tight compartment quite separate from production and processing was an organisational fallacy. As controls were developed, both in equipment and methods, operators and supervisors were having less and less to do except to look after quality. Psychologically, he thought it a great mistake to consider divorcing quality from the work of the operator, although it was of course necessary to have an umpire who could say at the end whether something was right or wrong.

Little was said in the papers about dimensions, and it must be admitted that it was particularly difficult to measure, and therefore to control, the dimensions of soft materials. Techniques which involved the use of high localised pressures were not suitable. There were, however, other means which could be adopted, and he felt that as production metallurgists, they could not expect to control the whole of their processes if they dodged one particular item which seemed to be of special importance.

MR. BENGT. STROM (Assistant Chief Metallurgist, A.B. Svenska Metallverken, Vasteras, Sweden), said that they had had a set of vacuum annealing furnaces installed in the works for about six months, and had found that while the critical temperature of sticking was increased by approximately 50° C. or more, it was not entirely eliminated. One of the drawbacks of this type of furnace was the lack of convection for the cooling cycle. On the question of the recrystallisation temperature of copper wire, MR. STROM said they had been supplying to one of the Swedish electric companies copper wire for enamelling purposes with a specified maximum recystallisation temperature.

MR. P. C. MURPHY (Research Metallurgist, Northern Aluminium Co., Ltd.), referring to the abnormal grain growth in aluminium wires by heavy drawing, said it could be regarded as an ordinary example of secondary recrystallisation. With high degrees of cold work, usually about 90%, there was a further peak in grain size which, depending on a number of factors, could be pushed back appreciably, so that it was not surprising some rather unusual results were obtained. On the

measurement of grain size by the Erichsen indentation test, using comparison with arbitrary standards, Mr. Murphy said such standard domes could conveniently be prepared in thoroughly homogeneous 1¼% manganese alloy which had been given a variety of cold reductions followed by different rates of heating to annealing temperature. Such standards could be used in assessing " orange peel " on all alloys and tempers, but correlation of actual spacial grain size was limited to alloys, gauges and tempers approximately similar to those in which the standards were prepared.

There was a real need for a generally accepted method of grain size measurement. The most complete method of specifying grain size was by giving the actual (that is, the spacial) average dimension of grains in each of three directions, describing, if necessary, any changes through the section. In practice, the determination of the dimensions need not be particularly tedious, as it could be shown statistically that 43% of the total number of grains in an aggregate would have a planor (as measured on the microscope section) grain size of at least 90% of the spacial grain size. By intelligently measuring ten of the larger grains along three axes, reproducible results on spacial grain size could be obtained.

Messrs. Field and Salter had referred to pits and cracks as defects encountered in tube drawing, especially of strong alloys. Such defects could, to a large extent, be associated with the extruded bloom and its subsequent treatment, and it was essential that bloom should be extruded at least as carefully as section. Most alloys required bloom annealing, and where possible the process should be carried out under conditions different from those used for intermediate annealing, as for adequate softening substantially higher temperatures were required, carrying with them the penalty that relatively slow cooling rates were required with most heat-treatable alloys. The optimum conditions for maximum softening might not always be desirable from other points of view.

Annealing represented another point where the tube was particularly susceptible to damage in handling. In the production of tubes in commercial purity aluminium and the 1¼% manganese alloy, which were not bloom-annealed, special care was necessary with the control of grain size. Production of shaped tubing called for special care in planning, if heat treatment was to follow the shaping pass, as the material must be soft enough to fill the die completely, but emerge with sufficient cold work to be immune from abnormal grain growth. The viscous compounded mineral oil used for tube and wire drawing could be replaced with advantage in certain applications by a spirit-dissolved wax which was applied by immersing the tube, draining it and allowing it to dry before drawing. Several passes could be made without further application, resulting in a considerably increased machine utilisation.

Mr. A. F. Norro (Superintendent Copper Smelter, Boliden Mining Company, Sweden), said that every charge of copper they made was tested for softening temperature, and there was a sort of mutual agreement that if it was higher than normal a special note was made of it. Mr. Miller had said that none of the commercial varieties of copper would have a softening temperature below 200° C., but he was proud to say that they had often had a temperature of 190° C.

Dr. K. Sachs, (Research Metallurgist, G.K.N. Group Research Laboratories) said he was intrigued by the test described by Dr. Betteridge and Mr. Cound for gas in nickel, in which they wrapped the nickel wire in glass, softened the glass and watched the bubbles. He wondered what gas content would be necessary to give an indication with other materials. The second question, addressed to Messrs. Edmunds and Lloyd, concerned the surface finish on dies. Was he right in thinking that hand polishing was a fairly substantial proportion of the total cost, and were dies polished to prolong the life or because the customer liked a shiny forging. He would also like to know on what basis one could assess the finish necessary from the point of view of die life, and whether it was possible to balance the cost of finishing the die against the customer's desire for a brightly-finished forging.

Mr. R. T. Thorley, (Aluminium Laboratories, Ltd.), stressed Mr. King's plea for standardisation in the methods of measurement of earing characteristics. The value quoted for the earing properties of a good material was a function both of the pressing conditions and of the method of measurement. Mr. Thorley put forward an expression based on $\frac{h_1-h_2}{h_2-k}$, where $h_1$ is the height of the ears, $h_2$ the height of the troughs, and $k$ a constant, which gave reasonably satisfactory agreement between sets of tools.

The statement by Messrs Field and Salter that a low-silicon, high-iron content was conducive to a fine grain size in commercial purity aluminium and the 1¼% manganese alloy, was queried by Mr. Thorley, as in their experience silicon between 0·2 and 0·5% had very little effect on the grain size.

In the paper by Messrs. Edmunds and Lloyd, there was no mention of a defect which could be very troublesome. It was found mostly in large forgings of the D.T.D. 683 type, and was most noticeable when present in the flash area. Fractures through the defects showed shiny facets, which they believed to be due to the presence of films of oxide. In the case of forgings made from extruded stock, the films could be introduced from the periphery of the extruded billet, and heavy scalping of the extruded stock would, therefore, be helpful. Such films could originate from the well-known sub-surface defects sometimes found in large diameter extrusions, but they could also be aggravated by the presence of defects in the surface layers of very large diameter C.C.P. billets which penetrated to a far greater depth than was generally realised.

Speaking for Professor von Zeerleder, Dr. E. A. Bloch, (Aluminium Industrie, A. G., Neuhausen, Switzerland) asked to what extent the molten lead bath referred to by Messrs. Field and Salter was used in the British aluminium industry. The same authors had stated that operating costs were higher with conveyor type furnaces than with batch furnaces. Was this due to higher labour and capital costs ? The annealing effect was improved by leaving the batch for an hour or so in the furnace to enable it to cool very slowly, but to assist production the load was often removed from the furnace and covered by an electrically heated bell. On the statement that the arbitrary Erichsen numbers corresponded with certain grain numbers, Dr. Bloch said that their experience indicated such correspondence to be found only with pure aluminium. In alloys, particularly the Al-Mg-Si type, the grain was always much greater.

Referring to the practice of interleaving sheets with

tissue paper during packing, MR. R. C. ANSELL (Northern Aluminium Co., Ltd.) said that the material cost something like ½d. /sq. yd., and could give rise to corrosion problems during certain conditions in transit and in storage. The problem could be dealt with in two ways. The acidity and chloride content of the paper as purchased could be controlled to an acidity of not more than 0·01% (calculated as sulphuric acid) and a chloride content of 0·25% (calculated as sodium chloride) for home use. For export material, where higher humidities might be encountered, these limits should be reduced by more than 50%. Alternatively, the paper could be treated with a corrosion inhibitor, but the cost would be increased thereby.

DR. SHILLING had mentioned thick plate used for complex machinings in which a porosity defect had been detected after machining. This defect was not true porosity, and was thought to be produced by gas retained in solid solution during casting diffusing to the grain boundaries, and passing into the areas where intermetallic constituents were being taken into solution: it only appeared after preheating. After casting, and before hot rolling, a slice was cut from each ingot, which was preheated, faced and treated with a penetrant dye used in conjunction with a developer solution. The plate was examined after heat treatment in a similar manner, but for comprehensive inspection ultrasonic methods were necessary.

A statement had been made that 520° C. was the maximum safe working temperature for nitrate salt baths, but it was common practice in the industry to operate baths at 535-540° C. for considerable periods, without any signs of trouble.

Referring to the heat treatment of complex aluminium bronzes to D.T.D. 197A, MR. R. E. BERRY (Manganese Bronze & Brass, Co., Ltd.) said that it was possible, in the course of treatments designed to bring the normal mechanical properties within specification, to cause precipitation of nickel and aluminium compounds, and so upset the fatigue and creep properties. In their experience it was the exception rather than the rule for such material to need heat treatment to meet the specification.

MR. WEBBER (Enfield Rolling Mills, Ltd.) said they had been very successful in steam-annealing copper strip at temperatures up to 650° C., such a high temperature enabling 8 tons to be annealed in a 10-hour shift. On the question of vacuum annealing furnaces, colleagues from Germany had expressed the opinion that they were cheaper to run than controlled atmosphere furnaces. He agreed with MR. BOND-WILLIAMS that the tool-room aspect of commutator bar production was of great importance.

## Authors' Replies

MR. MILLER said he was interest in MR. STROM'S remarks on the recystallisation of copper wire. A test at 200° C. had been mentioned and he wondered whether it was for an hour or a few minutes. German workers were investigating a test at 275° C. for 2 minutes, and in view of the interest in strand annealing and annealing in the enamelling oven a short time test was valuable.

In reply to DR. SACHS, MR. COUND, said that the glass test for gas in nickel alloys had been put more nearly on a quantitative basis, and had been discussed by Dr. Jenkins at a Birmingham meeting and subsequently reported in *Metal Industry*. Replying to the same speaker, MR. W. T. EDMUNDS said that dies were hand polished after the preliminary work had been done to ease the flow of the metal, and so increase die life.

MR. LLOYD said they agreed with MR. CHADWICK that the only way of differentiating with certainty between the different types of blister was by micro-examination. They had occasionally found oxide films which they presumed originated at the casting stage, but they did not always come to the surface at the flash. He would rather have expected oxide films to show up on a fracture as a dull patch, not, as in MR. THORLEY'S case, a light one. He would suggest two other possible causes: local overheating at the flash line; and stress corrosion failure.

MR. COLLIER thanked MR. BOND-WILLIAMS for his contribution on measurement, particularly of commutator bars. Edgewise curvature or bowing of strip, mentioned by MR. KEE, gave considerable difficulty, and it would always be necessary to have a tolerance for it, although a recent South African draft specification called for it to be zero.

In expressing appreciation of the various points raised, and the supplementary information given, MR. FIELD said the question of flatness was covered in D.T.D. 746, but practically nothing was bought to it. There was no particular difficulty in keeping down to ¼ in. in 10 ft. edge bowing with modern mills, although if 1 in. could be accepted, so much the better.

MR. BUSHELL had said that some metals were unreliable, but they were, on the whole the most reliable materials man had yet used. On the question of stretcher straightening, MR. FIELD said that the effect on the tensile properties of heat treated sheet was less than ¼ ton/sq.in. change in proof and ultimate stresses. In the matter of surface qualities, there were many in the industry, varying from sheet in which no blemish could be seen to grey and satin finishes. Similarly, various degrees of flatness prevailed according to subsequent use.

He agreed that silicon was not so important as iron in its effect on grain size, but before the value of high iron content was realised there was grave risk that too high silicon associated with low iron might result in increased grain size. The suggestion that there should be a generally recognised table of grain sizes was a good one. MR. MURPHY had referred to secondary recystallisation, and as much aluminium foil was reduced 90%, there was risk of grain growth with too high a temperature.

MR. FIELD concluded by assuring PROFESSOR VON ZEERLEDER that the lead bath referred to was purely experimental and was not used in commerce.

## Conclusion of Meeting

In bringing the symposium to a close, the chairman, MR. THOMAS, said they would all agree that the day's discussion, which could easily have continued for a much longer time, was a compliment to the authors, and there was no doubt that from such meetings all benefited. He was a little surprised that no reference was made to the ultimate control—the customer—because unless they could give him what he wanted at a price no greater than that at which he could get it elsewhere, they would be out of business.

MR. THOMAS concluded by extending the meeting's thanks to the authors of an excellent series of papers, and to DR. SINGER for the excellent way in which he had introduced them.

# Hard Soldering Aluminium and its Alloys

## Sheffield Smelting Company Developments

THE object of an investigation carried out in the laboratories of The Sheffield Smelting Co., Ltd., was the provision of an improved method of joining aluminium and its alloys, an essential requirement being that the joint should be corrosion resistant. Because of this, the mechanism of corrosion was studied in some detail, and corrosion testing formed a major part of the investigation. The outcome of this work was the development of the Thesscal process of hard soldering, so-called because it fills a gap between soft soldering at 250° C., or less, and brazing at about 600° C. Both these processes have drawbacks for certain applications, for soft soldered joints usually have low corrosion resistance and low strength, whilst brazing involves temperatures which are too high for joining thin gauge sheet of the strong age-hardenable alloys such as duralumin, and also introduces risk of distortion.

Two solders were developed as a result of this work: Thesscal A and Thesscal P.12, with melting ranges of 420–450° C. and 440–510° C. respectively. The former is a free-running solder, and is used with Flux No. 92 on sheet metal and fabricated forms of aluminium, whereas the P.12 solder is not so free running, can be moulded in much the same way as plumber's solder, and is used for the repair and joining of castings. They can also be used for joining aluminium and its alloys to copper and nickel, and their alloys, mild steel and even stainless steel. After extensive tests, at the Royal Aircraft Establishment, Farnborough, of the corrosion resistance of soldered joints between aluminium and various aluminium alloys, these solders have been approved by the Ministry of Supply, Thesscal A to D.T.D. 900/4367 and P. 12 to D.T.D. 900/4398. They are also approved by the Air Registration Board.

Besides the R.A.E. tests, soldered joints have been subjected to well known standard accelerated tests used by industrial laboratories, and by the Aluminium Development Association, for the assessment of corrosion resistance. Salt spray tests have been made by the Ministry of Supply and by a number of industrial research laboratories. Humidity tests lasting nine months have also been made at the Radar Research Establishment, Malvern. Finally, sets of soldered joints have been exposed outside in the industrial atmosphere of Sheffield for more than two years, and the joints are still mechanically sound.

### Joining with Thesscal A

For copper, brass, steel, nickel and commercially pure aluminium, clean sheet and machined surfaces normally need no preparation before soldering, but with aluminium alloys with a total alloy content exceeding 3%, mechanical cleaning is generally advisable.

Any normal combination of flame gases can be used for the heating torch: a neutral flame produces satisfactory results but this factor is not critical, and slightly oxidising or reducing flames can be used safely. Particularly when working with thinner gauges of metal, the technique of only playing the flame between applications of flux and solder is recommended, so that the latter act as their own temperature indicators. The size and composition of the flame should, therefore, enable the operating temperature to be reached and then maintained with intermittent flame application.

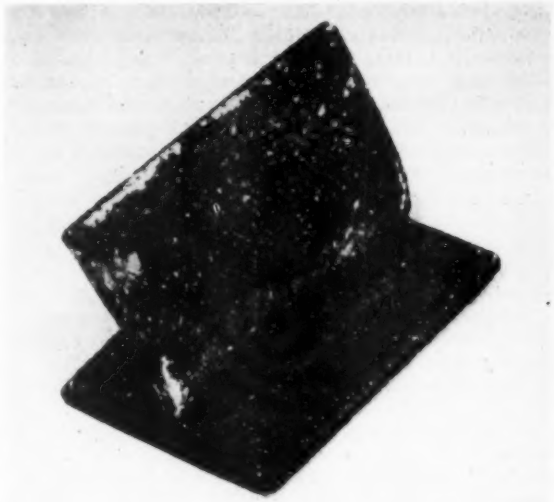

**Fig. 1—A joint between two sheets of aluminium after two years' exposure in the industrial atmosphere of Sheffield. The joint is still intact after bending.**

Mass production techniques of Thesscal hard soldering, such as electro-gas, furnace, high frequency, dip, and salt bath, are being investigated and to this end experiments are being carried out by the Sheffield Smelting Co., with the co-operation of industrial users.

Thesscal A solder, in the as-cast form, has a U.T.S. of 9·4 tons/sq. in., an elongation of 5%, and an electrical conductivity of 22·3% I.A.C.S. The strengths of typical joints are given in Table I.

Thesscal Flux 92, is a powder with a melting point of approximately 400° C. If it is required in paste form,

TABLE I.—JOINT STRENGTHS.

| Joint Combination | Shear | | Tensile | |
|---|---|---|---|---|
| | Location | Strength tons/sq. in. | Location | Strength tons/sq. in. |
| Aluminium to Aluminium | In aluminium | 4·2 | In aluminium | 5·1 |
| Aluminium to Steel | — | — | In aluminium | 5·1 |
| Aluminium to Copper | — | — | In joint | 3·7 |
| Al-2% Mg to Al-2% Mg | Part in joint, part in alloy | 7·2 | In joint | 7·3 |
| Duralumin to Al-2% Mg | In Al-2% Mg | 8·8 | — | |
| Duralumin to Duralumin | Mainly in duralumin | 9·12·5* | In joint | 8·11* |
| Duralumin to Copper | In joint | 7·1 | In joint | 4·9 |
| Duralumin to Steel | Part in duralumin, part in joint | 7·9 | In joint | 7·3 |
| | | 7·9 | | |

* Dependent on aged strength of duralumin.

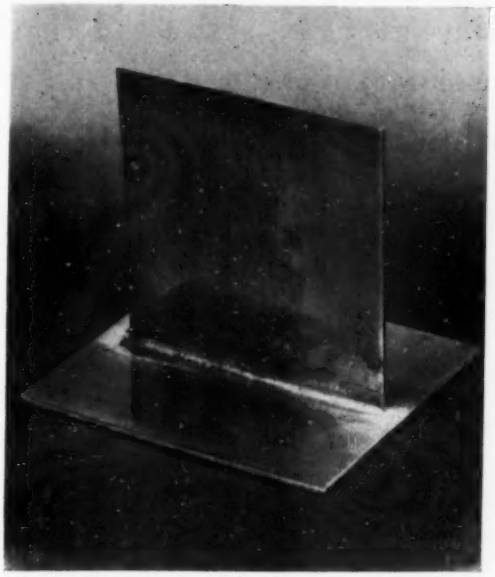

**Fig. 2—Note the neat joint line on duralumin to aluminium joint made with Thesscal A on $\frac{1}{16}$ in. sheet.**

**Fig. 3—The same joint as that in Fig. 2 after being hammered. The aluminium parent metal has cracked but the joint line is completely unaffected.**

it can be mixed to a creamy consistency with (*a*) water containing a suitable wetting agent, or (*b*) a mixture of one part methylated spirits to three parts water. Both these pastes are non-inflammable and will spread on surfaces which are clean but not degreased.

### Applications

The field of application of the Thesscal process is very wide. A complete range of aluminium alloys can be joined successfully, including aluminium-manganese, aluminium-magnesium, duralumin, and casting alloys including L33. These alloys can be joined to one another and to copper, brass, phosphor bronze, tungum, gum metal, nickel, nickel silver, steel and stainless steel.

Because of its relatively low operating temperature, many joining operations between aluminium components can be successfully carried out which would be rather tricky with higher temperature processes. For instance, one important use is for equipment, and for unstressed or lightly stressed parts in aircraft where thin sheet and sections of duralumin can be joined by unskilled labour without fear of fusion.

The fact that dissimilar metals can be joined successfully opens up new possibilities for designers in their aim to produce stronger and lighter components. The process also allows the building up of components in aluminium along lines similar to those in which brass components are built up using silver solders.

Again, as a result of the relatively low operating temperature, the joining of industrial aluminium-magnesium and aluminium-silicon alloys is facilitated.

### Repair and Joining of Castings

Broadly speaking the type of work for which the Thesscal process is suited in the casting field includes: (a) filling holes or other flaws which may be seen on the casting surface, or which may appear only after costly machining; (*b*) filling holes which have been incorrectly drilled; (*c*) retapping filled holes with suitable screwed threads; (*d*) building up lugs or other protrusions on the surface of the castings, or over holes; (*e*) joining castings or parts of castings together. The range of materials which can be treated is very wide, and includes all the alloys from LM-0 to LM-20 in B.S. 1490.

### Precautions

There are two considerations to be taken into account when using the process. The first is that, as is the case with silver soldering, the flux is corrosive, and must be washed off the joint. The Thesscal flux, however, is extremely soluble in hot water, much more so than silver soldering flux.

The second consideration is that Thesscal soldered joints cannot be anodised because they are attacked by the anodising solution. This can be overcome by using proprietary stop-off lacquers prior to anodising, but where some form of protection of the whole assembly is called for by specification, etching primers and paints can be used quite successfully. This alternative method is approved by the Ministry of Supply.

## Long Service Dinner

On Friday, April 1st, the Derby Works of International Combustion, Ltd., was the venue of a dinner and entertainment to over 100 members of the staff who have completed 20 years service, or more, with the organisation. The toast "The 'Old Campaigners' and the Company" was proposed by Ald. A. Sturgess, O.B.E., J.P., member of the East Midlands Electricity Board, and replied to by Sir Bernhard Binder, F.C.A., director of International Combustion (Holdings), Ltd. This is the second long service dinner held at Derby—at the first, held in 1949, there were 372 employees present to celebrate 20 years service.

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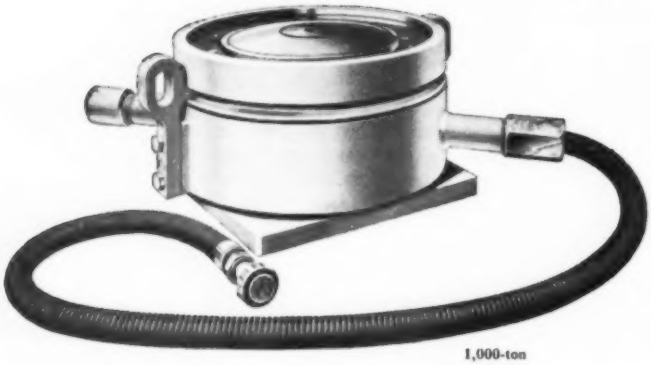

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# NEWS AND ANNOUNCEMENTS

## Joint Metallurgical Societies' Meeting in Europe

THE American Institute of Mining and Metallurgical Engineers and the American Society for Metals have accepted invitations from the Iron and Steel Institute and the Institute of Metals and from the leading metallurgical societies in Belgium, France, Germany and Sweden to send representatives to a Joint Metallurgical Societies' Meeting in Europe. The dates of the meetings are June 1st to 7th in Great Britain; June 9th to 12th in Dusseldorf; June 13th at Liege; June 14th to 18th in France.

The objects of the meeting are to establish personal contacts between experts in the U.S.A. and Canada and in Europe; to provide opportunities for scientific and technical discussion; to enable experts from the U.S.A. and Canada to see something of the metallurgical industries of Europe; and to foster better understanding and co-operation among all concerned.

The meeting will cover iron making (including coke and ore preparation); steel making; mechanical working and heat treatment, both ferrous and non-ferrous (including hot and cold rolling, forging, wire drawing and continuous casting, but not the foundry industry); refining and fabrication of non-ferrous metals; heat treatment; surface treatment (including finishing and coating); metallurgical education and training in industry; and research.

## Second Summer School in Corrosion

A SUMMER School will be held at Battersea Polytechnic from the 19th to 22nd July, 1955, the topic being "Oxidation of Metals." The panel of lecturers will include Dr. W. Betteridge (Mond Nickel Co., Ltd.), Dr. T. P. Hoar (Cambridge University), Dr. I. Jenkins (G.E.C.), Dr. O. Kubaschewski (N.P.L.), Mr. R. L. Samuel (Diffusion Alloys, Ltd.), Mr. H. T. Shirley (Brown-Firth Research Laboratories), and Mr. J. Stephenson (U.K. Atomic Energy Authority). The following aspects of the subject will be considered: theoretical principles of dry corrosion and anodic oxidation; oxidation and scaling of heat resisting steels; alloys for electrical heating elements; nickel-base alloys for high temperature use; oxidation resistance of the rarer metals; diffusion coatings for resistance to oxidation; and the effect of gases on high temperature oxidation. In addition to the lectures there will be discussions on the theoretical and practical aspects of oxidation, a demonstration and exhibition, a film show and a works visit.

The fee for the course will be 3 guineas, and accommodation during the course is available at the Polytechnic Hostel, if required, for an inclusive charge of 5 guineas.

## Plant Engineers' Conference

THE theme of the 8th Annual Conference of Incorporated Plant Engineers, to be held at the Hotel Majestic, St. Annes-on-Sea, from May 18th-20th, will be set by an address on "Plant, People and Prosperity", to be given by Mr. E. E. Butten, Chairman of Personnel Administration, Ltd. The Conference programme provides also for four Discussion Groups on subjects widely separated in nature but of equal interest and value to all sections of industry at the higher executive level. The subjects to be discussed by these Groups are "Electrical Safety in Factories"; "Can Work Study and its Incentives be applied to Maintenance?"; "Is there a Future for Tracked Earth Moving Plants in view of the Developments in Rubber Tyred Earth Moving Plants?", and "The Impact on the Plant Engineer of New Engineering Materials".

## Metal Finishing Conference

THE Annual Conference of the Institute of Metal Finishing will be held this year at Torquay from May 17th to 21st, when the following papers will be presented for discussion:—

*Wednesday* (9.30 a.m. to 12.30 p.m.).
Williams and Hammond, "The Effect of Chromium Plating on the Fatigue Strength of Steel"; Fry, "A Study of Cracking in Chromium Deposits"; Pick, "A New Method of Analysing the Stresses and Strains in Deposited Coatings".

*Wednesday* (2.30 p.m. to 4.30 p.m.).
Jepson (Miss), Meecham and Salt, "The Electrodeposition of Iron-Zinc Alloys"; Hoar and Bucklow, "On the Electrodeposition of Tungsten-Cobalt Alloys from Aqueous Solutions"; Britton and De Vere Stacpoole, "Corrosion Tests of Electrodeposited Tin-Cadmium Coatings on Steel".

*Thursday* (9.30 a.m. to 12.30 p.m.).
Hickling and Rostron, "The Chemical Polishing of Mild Steel by Hydrogen Peroxide-Oxalic Acid Mixtures"; Clarke and Andrew, "The Electrolytic Oxidation of Copper and Copper Alloys in Hot Alkaline Solution"; Wilman, "The Structure and Growth of Electrodeposits, and the Modifications caused by Brightening Agents".

*Friday* (9.30 a.m. to 12.30 p.m.).
Wallbank and Layton, "The Barrel Plating of Screw Threads"; Walker, Bentley and Hall, "Electroforming in Electronic Engineering".

*Friday* (2.30 p.m. to 4.30 p.m.).
Smith, "The Electron Microscope in the Study of Paint"; Nicholson and Jenkins, "The Blistering of Paint Films on Steel Surfaces".

*Saturday* (9.30 a.m. to 12 noon).
The Rt. Hon. the Earl of Halsbury, "Metal Finishing—Technique or Technology", introducing Discussion on "Education for the Metal Finishing Industry".

Excellent arrangements have been made for the social side of the meeting, including a programme for the ladies attending the Conference and the competition for the John Preston Golf Trophy.

## Non-Destructive Testing Meeting

The Summer Meeting of the Non-Destructive Testing Group of the Institute of Physics, being arranged jointly by the Group and the Scottish Branch of the Institute, will be held in the Royal Technical College, Glasgow, from June 22nd to June 25th, 1955.

Of the nine papers to be read, one will deal with teaching methods ("Training for Radiography" by Mr. T. S. Wiley); and two with physical theory ("Surface Properties: Their Possible Use in the Testing of Materials", by Dr. R. N. B. Stephens, and "The Physical Meaning of Indentation and Scratch Hardness",

by Dr. D. Tabor). The remaining papers include "Shipyard Radiography", by Mr. E. J. Duffy; "Non-Destructive Testing in the Boiler Making Industry", by Mr. W. G. Marskell; "The Assessment of Ultrasonic Techniques", by Mr. H. L. Carson; "Problems in the Interpretation of Ultrasonic Tests on Steel", by Messrs. I. M. Mackenzie and R. Kennedy; and "Neutron Radiography", by Dr. J. Thewlis and Mr. R. T. P. Derbyshire. The meeting will conclude with a Discussion Group opened by Dr. E. G. Stanford, with a short paper on "The Scope of Non-Destructive Testing", in order to review the research work which will be required to promote progress in the field of non-destructive testing.

## Exide South African Factory

A £1,000,000 battery factory—the largest in Africa and one of the best equipped in the world—is now in production at East London. Owned by The Chloride Electrical Storage Co., S.A., (Pty.), Ltd.—an associate of the Manchester firm of Chloride Batteries, Ltd.—the new plant is making the well-known Silver Exide car battery. The light, airy and spacious buildings of the new factory cover 120,000 sq. ft. of a five acre site.

# Personal News

MR. J. P. A. MELDRUM and MR. R. H. S. TURNER have been appointed Directors of Metropolitan-Vickers Electrical Co., Ltd. Mr. Meldrum has been Manager, Home Sales, since January 1955; Mr. Turner was appointed Works Manager, Trafford Park Works, in April, 1954.

MR. T. LAND, Managing Director of Land Pyrometers, Ltd., Sheffield, presented a paper on "New Pyrometers for Glass and Other Surfaces" at Institut National Du Verre at Charleroi, in Belgium, on April 15th.

MR. F. A. KING and MR. A. J. HUGHES formally retired from the Board of Kelvin & Hughes, Ltd., at the end of March, after some 60 years of service with the company.

MR. F. W. BATES, who first joined the company as an apprentice in 1927, later becoming General Works Manager, has now been appointed to the Board.

MR. E. J. BATCHELOR has been appointed General Manager of the Automobile Division of The David Brown Corporation, Ltd., the manufacturers of Aston Martin and Lagonda cars.

DR. H. K. HARDY has been awarded the degree of D.Sc. (Eng.) by the University of London. Dr. Hardy's researches on the mechanism of precipitation, on the effect of trace elements on precipitation, and on the application of thermodynamics to phase constitution, have been carried out at the Fulmer Research Institute whilst occupying the posts of Head of the Physical Metallurgy Section and Senior Metallurgist.

MR. J. E. GAMAGE and MR. J. R. SPANSWICK have been appointed Directors of Electric Furnace Co., Ltd.

MR. E. F. WATSON has been appointed a Director of Electric Resistance Furnace Co., Ltd.

SUNVIC CONTROLS, LTD., announce that they have appointed MR. D. M. GILL as Technical Representative in the North West England Area for their Process Control Division. MR. F. N. YATES of 36a, Brownville Road, Heaton Moor, Stockport, will be their Technical Representative in the same area for the Scientific and Industrial Divisions. The corresponding appointments for their London and Home Counties Area will be held by MR. F. C. CHAVENTRE and MR. W. F. DORMAN.

MR. J. H. EVANS has been appointed Chief Metallurgist of the British Timken Organisation.

MR. W. SCOTT, Managing Director of Armstrong Whitworth (Metal Industries), Ltd., and Jarrow Metal Industries, Ltd., has just returned from a business tour of the United States, where he met executives of several important American companies.

MR. H. LLOYD, Export Director of Marsh Brothers & Co., Ltd., Sheffield, is at present on an extended business tour, in the course of which it is planned to spend a fortnight in Ceylon, six weeks in Australia and four weeks in New Zealand.

MR. E. G. SNELUS, M.B.E., has been appointed Sales Manager at the Birmingham Metal Works of McKechnie Brothers, Ltd.

MR. M. SELIGMAN, Managing Director, and MR. J. F. B. JACKSON, Deputy Managing Director of A.P.V.-Paramount, Ltd., now one of the largest producers of stainless steel castings in the country, recently returned from a visit to the leading Swiss stainless steel foundries.

MR. E. AUSTYN REYNOLDS, who has been a well-known figure in the aluminium industry for the past twenty years, has recently resigned his position as Director of Development with T.I. Aluminium, Ltd., for personal reasons. Joining the family firm of Reynolds Tube Co., Ltd., in 1922, Mr. Reynolds turned his attention to aluminium in 1933 and for many years continued to expand the aluminium side of Tube Investments, Ltd. Many will remember his term of office as President of the Aluminium Development Association which, coinciding with the Festival of Britain, made considerable demands on his energy.

MR. J. E. CALVERLEY has retired from the English Electric Co., Ltd., where he was formerly Deputy Chief Engineer (Technical). Since January 1952 he has been engaged on special consultancy duties for the Company.

# Obituary

WE regret to record the deaths of the following:—

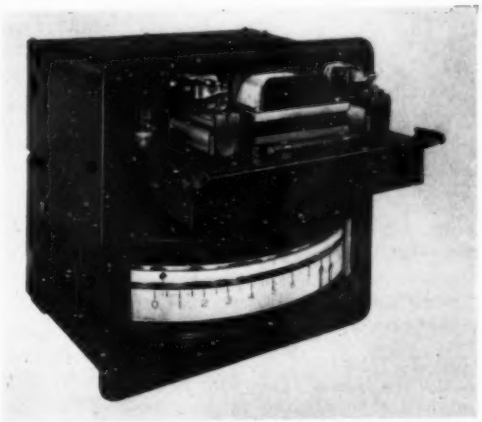
MR. J. L. SMITH, Managing Director of The J.L.S. Engineering Co., Ltd., makers of heat treatment furnaces and ovens, who died suddenly on March 28th, 1955. Under Mr. Smith's guidance the company established a high reputation in its specialised field.

MR. K. C. GARDNER, Chairman of United Engineering & Foundry Company, of Pittsburgh, Pa., died at his home at Coriapolis Heights on April 15th. Mr. Gardner was closely associated with the engineering design of Britain's strip mills in the new steelworks of Wales. His company's designs were used by the British and American manufacturers for the construction of the first strip mill at Ebbw Vale, shortly before the war, and also at Margam, Trostre and Velindre, where the most modern tinplate works in Europe have recently been built. His death is a great loss to his many friends here, but the impressive Welsh works remain as a monument to his enterprise as an outstanding American engineer and a good friend of Britain.

# RECENT DEVELOPMENTS
## MATERIALS : PROCESSES : EQUIPMENT

### Temperature Measurement and Control Instruments

INTERESTING introductions in the Electroflo range of instruments for temperature measurement and control are the latest model 156B Electronic Indicating Control Pyrometer and the Model 103/156 Recording Controller. The former is an improved version of the instrument which has proved its efficiency and reliability in many installations. It can be calibrated to any standard range between 0 and 3,000° F. or 0 and 1,600° C., and consequently has a wide sphere of usefulness in the temperature control of electrically-heated or fuel-fired furnaces: it can be provided for 2- or 3-position control to suit different systems of heating. An important feature of this instrument is that only one standard type thermionic valve and its associated circuit is required to initiate control action to hold the temperature constant within very close limits. The indicating and control units are both contained in a compact cabinet, finished grey and suitable for either flush panel or wall mounting.



**Model 156B electronic indicating control pyrometer.**

The recording controller combines the Model 156B described above, with the Series 103 Recording Pyrometer. With this instrument a record can be made of temperatures at one or three points with control of temperature at one point. The instrument can alternatively incorporate an alarm system—audible and/or visible—to operate in the event of any of the recorded temperatures exceeding a pre-determined maximum.

*Electroflo Meters Co., Ltd., Abbey Road, Park Royal, London, N.W.*10.

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### Darkroom Safelights

RECENT improvements in the design and finish of these two well-known Kodak safelight lamps have resulted in the introduction of new models, which while retaining the qualities of their predecessors are at the same time stronger in construction, and lower in price. The Kodak universal safelight lamp, which accommodates either one or two safelights at will, is a versatile model which can be made to give direct overhead illumination, indirect (ceiling) illumination, or combined direct and indirect illumination through both safelights simultaneously. The Kodapan safelight lamp provides a diffused indirect illumination throughout a darkroom used for negative development, particularly where fast or extreme-speed types of panchromatic material are to be handled.

Improved features of the two safelight lamps include the following: the metal lamphouses are spot-welded for greater strength and are treated so as to be rust-proof; flat springs are fitted into the grooves into which the safelight slides and these springs press the safelight against the outer flange and prevent light leak. Both safelight lamps are now finished externally in hammered grey stove enamel and internally in white. A 25-watt bulb is recommended for use with either of these safelight lamps.

*Kodak Ltd., Wealdstone, Harrow, Middlesex.*

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### Concrete Floor Paint

A COLOURED rubber paint specifically formulated to meet the demands of industrialists who wish to protect and decorate new and old concrete floors, has recently been introduced by Allweather Paints, Ltd. Its use on concrete floors eliminates dust, and being alkali-resistant this paint can even be used on 14-day old concrete floors without fear of breakdown. Its tough rubber film resists hard wear and since the paint is very quick drying the freshly painted floor is out of commission for only a very short spell. Known as Pitan Concrete Floor Paint it is available in a wide colour range, and the manufacturers recommend the application of one (dark shades only), two or three coats to provide a dust free surface which can be repeatedly washed without deterioration.

*Allweather Paints, Ltd.*, 36 *Great Queen Street, London, W.C.*2.

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### Quick Response Féry Telescope

IT is sometimes desirable to employ a radiation temperature measuring instrument in conditions where it is essential to obtain a quick reading of the temperature. The Cambridge quick response Féry radiation pyrometer which has been developed for this purpose, incorporates improvements in electrical and mechanical details. Normally, the rate of response of the instrument is such that 99% of a radiation input corresponding to a full scale reading will be measured in 4·6 seconds, and 99·9% in 6·9 seconds. Where a still quicker response is required for exceptional applications at moderate temperatures, a specially fast receiver can be provided to give 99% full scale response in 1·2 seconds and 99·9% in 1·8 seconds.

The measuring element is a thermocouple, which is

**Quick response radiation pyrometer.**

heated by radiation from the hot body, and a compensating device is introduced into the electrical circuit to reduce the effect of change in ambient temperature (e.g., about 1° low for 1° ambient rise at 1,650° C. and 10 mV. output). Provision is made for water cooling, however, to ensure accurate readings, and an alarm may be fitted to give warning of failure of the water supply. The shortest normal distance of operation, measured from the front face of the instrument, is 3 ft., and the size of hot area must not be less than about $1\frac{3}{4}$ in. for every 3 ft. distance. This size is just sufficient to ensure that the image of the hot body when viewed through the telescope and properly focused will completely cover the aperture surrounding the sensitive element.

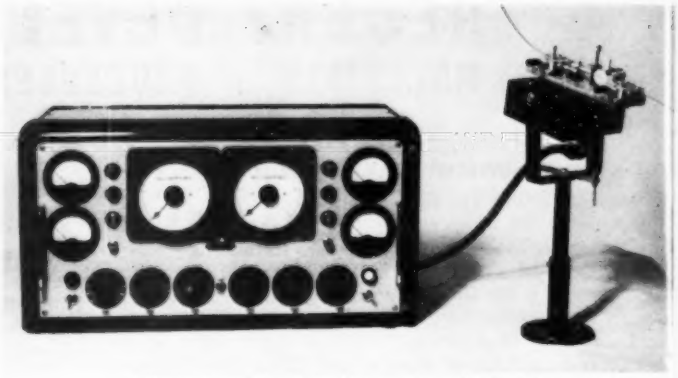
The instrument can be used in conjunction with suitable indicators and recorders, electronic recorders being recommended to enable full advantage to be taken of the fast rate of response.

*Cambridge Instrument Co., Ltd.*, 13, *Grosvenor Place, London, S.W.*1.

## Dimensional Limit Indicator for Rolled Strip

THIS instrument has been designed to give an instantaneous indication of any variation in the dimensions of metal strip during the final drawing process. It gives an immediate aural and visual warning of changes outside predetermined limits and, if required, can be made to stop the mill.

The control unit has duplicate indicators and setting controls for width and thickness measurement. The smaller meters show all dimensional variations (the upper one positive, the lower one negative) and the tolerance required can be pre-set by individual controls. Green lights indicate whether the material is within the specified limits. Variations outside the tolerance limits are indicated by a red light (positive) or an amber light



**Dimensional limit indicator for rolled strip.**

(negative), accompanied by a buzzer warning signal. A " break " contact is available to break the mill contactor circuit.

The micrometer unit, connected to the control unit by a flexible cable, is of small dimensions for ease of fitting where there is little room between the die or rollers and the take-up drum. Ball bearing guides accommodate the strip and the assembly can be mounted to give an adequate float where necessary as the take-up drum fills. The essential electronic components are housed in a totally enclosed case with adjusting controls readily accessible.

The original unit was designed for inspecting strip approximately $\frac{3}{16}$ in. wide by 0·030 in. thick, but with suitable guides there is no limits to the dimensions of the strip which can be accommodated. Great sensitivity can be obtained with this equipment and tolerance limits can be set within a range of ± 0·0003 in. to ± 0·01 in.

*Sutton Coldfield Electrical Engineers, Ltd., Clifton Street, Sutton Coldfield, Warwickshire.*

## Fungicide to Combat Paint Discoloration

RECENT surveys have shown that the discoloration of paint and the failure of the film, particularly in buildings of high humidity, is often due to attack by fungus rather than ordinary dirt. The Nuodex organisation, research pioneers in this field, has isolated as many as 30 different fungi which flourish on paint films, and this work has resulted in the introduction of a new fungicide, Nuodex 321.SS, which is completely soluble in mineral spirits and compatible with emulsion paint systems, and effectively precludes such premature decorative loss of the paint. The Nuodex fungicide has shown its value in numerous industrial establishments in which high humidity conditions presented problems of painting costs.

It has been found that some paint formulations actually helped mould growth, the paint serving as a source of food for the fungi. This necessitated the study of individual ingredients in paint compounds and the influence each had on mould susceptibility. The ideal antifungus paint is therefore formulated accordingly and reinforced by an effective fungicide. Nuodex fungicide is permanent and insoluble in water and will neither evaporate nor crystallise from the paint film on exposure. It will remain fully effective for the service life of the paint.

*Nuodex, Ltd., Birtley, Co. Durham.*

LABORATORY METHODS
MECHANICAL · CHEMICAL · PHYSICAL · METALLOGRAPHIC
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# Metallurgical Analysis: Some Physico-Chemical Considerations of Precipitation Reactions and Electrolytic Separations

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*Chemical analysis, whether qualitative or quantitative, is based on fundamental physico-chemical principles, which are, perhaps, not so widely understood by practising analysts as they might be. In this article, based on a paper presented to the Analysis Group of the Sheffield Metallurgical Association in November, the author considers two important aspects of particular interest to the metallurgical analyst.*

IT has been customary in the past for chemistry students to devote a large proportion of their practical work to chemical analysis. Very soon the tyro finds himself treading the well worn path of group analysis, precipitating some sulphides in acid solution and others in alkaline, and it is considered that practice at this sort of work is an important contribution to his understanding of chemistry. Undoubtedly this would be so if the student were helped to apply quantitative theoretical considerations to his practical achievements, but all too frequently he is brought up to regard his practical work as a thing apart from his theoretical studies. As a consequence, the student and, indeed, very many skilled analysts have little knowledge of the underlying principles of qualitative or quantitative analysis, and are ignorant of such questions as, for example, why do some sulphides precipitate in acid solution and some in alkaline. It seems worthwhile, therefore, to investigate some of these underlying physico-chemical principles and, because of limited space, two aspects of chemical analysis have been chosen that are of considerable interest to the metallurgical analyst, viz., reactions involving the formation of precipitates such as sulphides, hydroxides, etc., and electrolytic separation techniques, e.g., the mercury cathode process often used in the determination of aluminium in steel.

## PRECIPITATION REACTIONS

### Law of Mass Action

Before discussing precipitation reactions it is necessary to give some attention to the laws underlying equilibria between ions in aqueous solution. As every chemist knows, the law of mass action first enunciated by Guldberg and Waage is of considerable help in assessing equilibrium conditions, but it should be appreciated at the outset that a considerable difficulty lies in obtaining quantities that are accurate measures of active masses. The law of mass action states that if a reaction such as

$$A + 2B = 3C + 4D$$

occurs, equilibrium conditions are given by

$$K = \frac{[C]^3\,[D]^4}{[A]\,[B]^2}$$

where $K$ is the equilibrium constant and [A], [B], [C] and [D] are the active masses of the reactants and products A, B, C and D. The active masses were at one time considered to be identical with concentrations, and the early success of the law using this postulate was undoubtedly due to the very dilute solutions studied. Under such conditions, interionic effects are small, and the active masses or "activities" of the ions are proportional to their concentrations. Consequently, when ionic concentrations are used in the mass action equation, $K$ is constant under equilibrium conditions for a particular temperature. If the same procedure is employed with more concentrated solutions, $K$ becomes variable, and this is fundamentally due to the fact that ionic concentrations are no longer measures of active masses or activities. Because of interaction between the ions, their chemical activities are no longer proportional to their concentrations, and it is necessary to multiply a particular ionic concentration by a factor $f$ (called its activity coefficient) to produce a quantity which is a measure of its active mass or activity. It is obviously important to be able to calculate $f$, and, fortunately, this is often possible as a consequence of the brilliant work of Debye and Hückel. Thus

$$\log f_i = -\frac{0{\cdot}505 Z_i^2 \mu^{0{\cdot}5}}{1 + 3{\cdot}3 \times 10^7 a \mu^{0{\cdot}5}} \qquad (1)$$

where $f_i$ = activity coefficient of the ion i,
$Z_i$ = charge number on the ion i,
$a$ = average effective diameter of all the ions, and
$\mu$ = ionic strength of the solution defined by $\mu = 0{\cdot}5 \Sigma C_i Z_i^2$ ($C_i$ = gram-ions/litre of ion i).

For very dilute solutions (1) reduces to

$$\log f_i = -0{\cdot}505 Z_i^2 \mu^{0{\cdot}5}$$

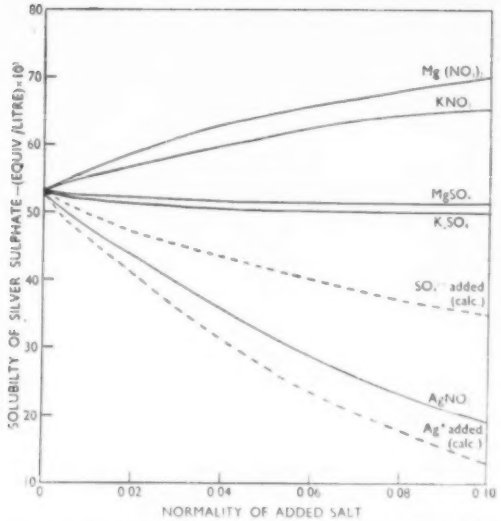


**Fig. 1.—Influence of various salts on the solubility of silver sulphate.**

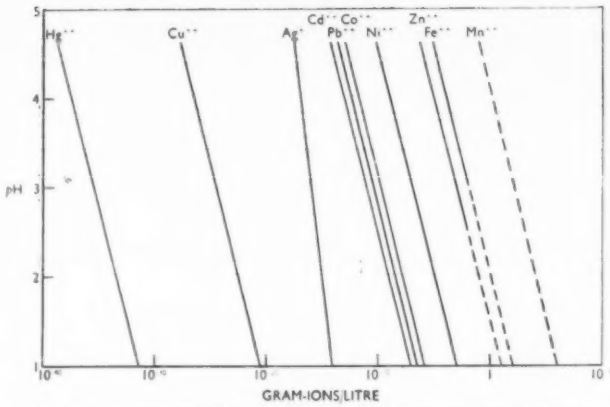


**Fig. 2.—Concentrations of ions in aqueous solution in equilibrium with the respective sulphides and hydrogen sulphide at 1 atmosphere pressure against pH values.**

and when used in this form it is unnecessary to know the average effective diameter of all the ions. An important practical conclusion from this equation is that the presence of large numbers of ions in solution, especially if they have high charge numbers (i.e., a solution of large ionic strength) produces low values for $f$; as a consequence, their activities are lowered, causing, as will be shown later, increased solubility of precipitates. Here is an important reason for keeping the concentrations of salts low during precipitations; a precaution that is well known to practising analysts.

To summarise, under equilibrium conditions, and when the solution is dilute, the classical law of mass action holds approximately when ionic concentrations are substituted for active masses or activities. However, as the solutions become stronger, it becomes more necessary to calculate and employ the activity coefficients ($f$). In what follows it will be assumed that the solutions are very dilute and that, consequently, ionic concentrations are synonymous with activities.

## The Solubility Product

When a precipitate such as $M_2X$ is in equilibrium with a solution, the following conditions may exist:

$$\underset{\text{solid}}{M_2X} \rightleftharpoons \underset{\substack{\text{associated}\\ \text{in solution}}}{M_2X} \rightleftharpoons 2M^+ + X^{--}$$

Many other equilibria are possible: for example, $MX^-$ ions could be present, but it will suffice for illustration purposes to assume the above state of affairs. Using the law of mass action it is obvious that

$$[M^+]^2[X^{--}] = \text{a constant (at constant temperature)} \quad (2)$$

This constant is of course the solubility product, but it is important to emphasise that $[M^+]$, for example, does not necessarily represent the total amount of M in solution as found by ordinary chemical analysis since, as already stated, other combinations of M (e.g., $MX^-$) are possible. However, for very insoluble precipitates (i.e., under conditions where the concentrations of $M^+$ and $X^{--}$ are low) the proportion of M as $M^+$ is very large. Thus, consider the equilibrium

$$\underset{\substack{\text{associated}\\ \text{in solution}}}{M_2X} \rightleftharpoons 2M^+ + X^{--}$$

Assume that prior to dissociation, the solution contains 1 mole in $V$ litres of $M_2X$ only, and that $x$ moles are dissociated at equilibrium. Then

$$\frac{K\,(1-x)}{V} = \frac{4x^3}{V^3}$$

i.e.,

$$K\,\frac{(1-x)}{4x^3} = \frac{1}{V^2}$$

Obviously, as $V$ increases, i.e., as the dilution becomes higher, $x$ increases, i.e., dissociation increases. Hence, for very insoluble precipitates (where $V$ is large), the dissolved ions may be considered to be completely dissociated, and such intermediates as $M_2X$ (associated in solution) neglected.

From the solubility product equation (2), it is obvious that the precipitation of a salt such as a sulphide may be caused by:

(*a*) increasing the concentration (activity) of salt anions; or

(*b*) increasing the concentration (activity) of salt cations.

Both methods are used in analytical chemistry and Fig. 1, taken from Vogel, shows the influence on the solubility of $Ag_2SO_4$ of various ions. Its solubility is decreased by the addition of sulphate or silver ions, but not as much as would be expected from simple solubility product calculations (the continuous and dashed lines are measured and calculated solubilities respectively). The deviations between the calculated and measured solubilities are a consequence of the interionic effects already discussed; these reduce the activity coefficients of the ions, and the effect is well illustrated by the increased solubility of $Ag_2SO_4$ arising from the addition of ions other than $SO_4^{--}$ and $Ag^+$, i.e., when $Mg(NO_3)_2$ and $KNO_3$ are added.

The sensitivity of precipitation to $p$H is generally a consequence of (*a*) and (*b*) above. Thus, oxalates are more soluble in solutions of low $p$H. For example, calcium oxalate will precipitate when

$$[Ca^{++}]\,[C_2O_4^{--}] > \text{solubility product.}$$

The following equilibrium exists

$$2H^+ + C_2O_4^{--} \rightleftharpoons C_2O_4H_2$$

and the equilibrium constant is sufficiently high to result in most of the oxalate ions being converted to the undissociated acid (or some other complex) in dilute acid solution. Thus, as the $p$H decreases, $[C_2O_4^{--}]$ decreases until $[Ca^{++}][C_2O_4^{--}]$ is less than the solubility product; complete solution of the precipitate occurs.

Similar mechanisms account for the increase in solubility with acidity of phosphates, carbonates, sulphates, sulphides, hydroxides, complexes such as nickel glyoxime and many other compounds. All rely on a decrease of the acid ions by reaction with hydrogen ions, e.g., the formation of $HCO_3^-$ or $CO_2$, $HSO_4^-$, $H_2S$, etc.

It is of interest to pursue the above argument quantitatively, and some calculations have consequently been made concerning the precipitation of sulphides and hydroxides.

## Precipitation of Sulphides

The calculation of the solubility of CuS at various $p$H levels may be taken as an example. The normal technique for precipitating sulphides is to pass $H_2S$ through the solution, with the pressure of the $H_2S$ gas approximately 1 atmosphere. Thus, when CuS is precipitated under such conditions, the following equilibria (amongst others) are eventually attained.

$$\underset{\text{solid}}{CuS} \rightleftharpoons Cu^{++} + S^{--}$$

$$\underset{\text{1 atmosphere}}{H_2S} \rightleftharpoons 2H^+ + S^{--}$$

and these give rise to the equations

$$[Cu^{++}][S^{--}] = \text{solubility product} \qquad (3)$$

$$[H^+]^2[S^{--}] = Kp_{H_2S} = \text{constant at 1 atmos. pressure} \qquad (4)$$

The dissociation constant of $H_2S$ into aqueous $H^+$ and $S^{--}$ ions and the solubility product of CuS are known at any particular temperature. Using (4) and any particular $p$H value, $[S^{--}]$ can be determined and this, when substituted in (3), gives a value for $[Cu^{++}]$. It is thus possible, for any $p$H level, to calculate a value of $[Cu^{++}]$ in equilibrium with pure solid CuS and pure $H_2S$ gas at 1 atmosphere pressure. This calculation has been made for several sulphides and the results are graphed in Fig. 2. It is immediately obvious that the sulphide solubilities, as judged by the concentrations of the ions indicated, decrease with increase in $p$H, and this broadly explains the well-known group separations; the most insoluble sulphides are first isolated by precipitation in acid solution, followed by a later precipitation of the more soluble sulphides in alkaline solution. It should again be emphasized at this stage that the calculated concentrations of the metal ions presented in Fig. 2 only approximate to real values when the actual concentration is very low. The values for $[Cu^{++}]$, etc., are approximate activity values, and differ markedly from concentration values in solutions of high ionic strength. Moreover, the value of say $[Cu^{++}]$ does not represent the total copper in solution but only the amount as $[Cu^{++}]$; thus the calculations have not included other forms e.g. $Cu^+$ or $CuCl_3^{--}$ which must be added to the $Cu^{++}$ amounts to provide the total soluble copper concentration. One difficulty which arises in connection with these considerations is that a sulphide can possess more than one solubility product at a given temperature. The solubility product under conditions when a sulphide just precipitates can be different from that when the same precipitate just dissolves. From Fig. 2 it would appear possible to precipitate nickel and cobalt in distinctly acid solution: this does not normally occur in practice, although such sulphides as CuS can be heavily contaminated with nickel or cobalt, by co-precipitation. However, once formed, nickel and cobalt sulphides will not dissolve appreciably in acid solution providing the pressure of $H_2S$ in the system is maintained around 1 atmosphere.

## Variation of Solubility Product with Particle Size

Mention has not yet been made of the variation of the solubility product of a salt with its particle size. It has been established practically that the solubility of a salt is increased by adding it to the solution as a very fine powder. This effect is of great practical importance; it has been stated, for example, that the solubility of $BaSO_4$ is increased 80–90% when the $BaSO_4$ solid consists of particles of 0·1–0·2$\mu$ diameter. It is possible by a simple thermodynamic argument to obtain a relationship between the chemical activity of a substance and its state of division, it being assumed that the effect is a consequence of surface energy. The activity of a substance may be regarded as its vapour pressure, and if a reversible isothermal series of operations is considered in moving a small element of the substance ($dn$ moles) from a particle of radius $r$ to the massive salt (i.e., where the specific surface is so large as to remain constant when $dn$ moles are added or withdrawn), it is found that

$$dn\,RT\ln a = \sigma\,dA \qquad (5)$$

where $T$ = absolute temperature
$R$ = gas constant
$a$ = activity of the surface of the particle relative to the massive state
$\sigma$ = surface energy of the particle
$dA$ = change in surface area of particle due to addition or withdrawal of $dn$ moles

$dn$ and $dA$ can be eliminated by expressing them as functions of the radius of the particle $r$ and its specific volume $v$, in which case (5) reduces to

$$\ln a = \frac{2\sigma v M}{RTr}$$

where M = molecular weight

Fig. 3 shows the result of applying this calculation to the activity of gypsum ($CaSO_4.2H_2O$). The activity of the massive material is taken as unity (i.e., $a = 1$ for the standard state where $r = \infty$); as the particle size decreases the activity increases and, theoretically, becomes infinitely large at zero particle size. The solubility of gypsum should follow a similar relationship since, assuming complete dissociation of the gypsum in solution, and that all $Ca^{++}$ and $SO_4^{--}$ ions have arisen from gypsum,

$$[Ca^{++}] \propto a^{\frac{1}{2}}_{\text{gypsum}}$$

Although the general trend of the curve has been verified by experiment, it is doubtful whether the actual solubility increases as markedly as is shown in Fig. 3 for the very small particle sizes. Small particles are usually charged, and on any particle the surface charges are all of the same sign and, consequently, repel one another. This results in a tendency for the surface of the particle to expand in opposition to the surface tension forces, so that the charged particle behaves as though its surface tension (i.e., surface energy) were very low,

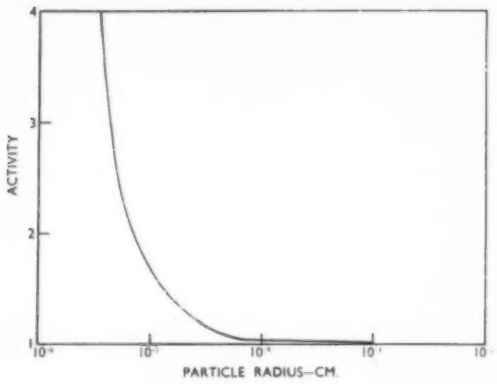


**Fig. 3.—Activity of solid calcium sulphate dihydrate at 25° C. against radius of particles (standard state = massive material).**

i.e., its solubility is not as far removed from that of the massive state as is indicated by Fig. 3.

Another reason for the breakdown of the relationship at low particle sizes results from the assumption that the surface energy is independent of particle size. The whole concept of surface energy, and, indeed, the thermodynamic treatment, relies on the particles containing large numbers of ions or molecules ; when these become small the particle cannot have a steady surface tension or vapour pressure.

The shape of the curve in Fig. 3 does throw some light on the problem of supersaturation. It suggests that if a clean solution is evaporated, salts will never deposit unless some nuclei are present of finite size (i.e., of $r > 0$). Obviously, the more concentrated the solution becomes, the smaller the nuclei can be to cause precipitation ; eventually the effective nuclei can be so small that their chance of formation is large and precipitation is assured. This theory explains reasonably satisfactorily the von Weimarn rule, which states that optimum conditions for coarse-grained precipitates occur when the precipitating reagent (e.g., $BaCl_2$) is added slowly. In this way heavy supersaturation is avoided and, consequently, only fairly large nuclei are effective in causing precipitation. The result is a precipitate consisting of a few large grains. If considerable supersaturation is induced by quick additions, much smaller nuclei are effective in commencing precipitation (see Fig. 3), and since the number of nuclei available increases as their size decreases, the final precipitate consists of many small particles, causing difficulties in filtration. Another practical aspect of this theory is that smaller particles, being more soluble than larger ones, will dissolve and reprecipitate on them. This is probably the reason for the well known fact that the filtering of $BaSO_4$ is considerably facilitated if the $BaSO_4$ is allowed to remain in contact with the solution for some time after precipitation.

## "Gatherers" and Complexing Agents for Sulphides

Reverting to sulphide precipitation, the fact that solubility is a function of precipitate particle size may possibly be the reason for the sluggishness exhibited by some metals, e.g., cadmium, in precipitating as sulphides. A well known technique for precipitating traces of a metal as sulphide is to add a " gatherer " to the solution, so that the trace metal sulphide is co-precipitated with or absorbed by " gatherer " sulphide. This follows logically from theory, since the condition at precipitation of, say, $M_2S$ are

$$[M^+]^2[S^{--}] = \text{constant} \times \text{activity of solid sulphide}$$

The fact that the solid trace metal sulphide activity is considerably reduced by solid solution in, or absorption by, the gatherer, results in $M^+$ ions leaving the solution. If only the trace metal were present, a much larger value for $[M^+]^2[S^{--}]$ would be required for separation, since the precipitate would consist of the pure solid sulphide of high activity.

Before leaving sulphides, the use of complexes for avoiding precipitation must be mentioned. To overcome, say, the precipitation of copper, at a constant acid $p$H level and in the presence of $H_2S$ at 1 atmosphere pressure (these conditions fix $[S^{--}]$), it is essential to reduce $[Cu^{++}]$ so that the solubility product given in (3) is not exceeded. A technique for accomplishing this is to add a reagent that will convert most of the $Cu^{++}$ ions to some other form. This can be achieved with $CN^-$ ions, the reaction being

$$Cu^{++} + \mathcal{E} \rightleftharpoons Cu^+ \quad [\text{e.g. } 2Cu^{++} + S^{--} \rightleftharpoons 2Cu^+ + S]$$

$$Cu^+ + 4\ CN^- \rightleftharpoons Cu(CN)_4^{---}$$

Because of the low instability constant of $Cu(CN)_4^{---}$ the $[Cu^{++}]$ is reduced to such a low value that CuS precipitation is avoided. On the other hand cadmium, although forming a complex :—

$$Cd^{++} + 4CN^- \rightleftharpoons Cd\ (CN)_4^{--}$$

is precipitated as sulphide. The instability constant of $Cd\ (CN)_4^{--}$ is large, so that the value of $[Cd^{++}]$ is little changed by the addition of $CN^-$ ions.

## Precipitation of Hydroxides

The laws governing the precipitation of hydroxides follow a similar pattern to the formation of sulphides. Two equilibria that can be employed in calculating hydroxide solubility are, in the case of iron, for example

$$\underset{\text{solid}}{Fe(OH)_3} \rightleftharpoons Fe^{+++} + 3\ OH^-$$

$$\underset{\text{nearly pure}}{H_2O} \rightleftharpoons H^+ + OH^-$$

These lead to the two equations

$$[Fe^{+++}][OH^-]^3 = \text{solubility product} \qquad (6)$$

$$[H^+][OH^-] = \text{a constant} \qquad (7)$$

and the similarity between these and (3) and (4) is apparent. At any $p$H level, $[OH^-]$ is fixed by the ionic product for water. Substituting the $[OH^-]$ value in (6) and using the appropriate solubility product, $[Fe^{+++}]$ may be determined. This calculation has been performed for a series of metal hydroxides and the results are graphed in Fig. 4. The results show the general increase in solubility that attends increase in acidity. It would appear that all the metals considered are precipitated at high $p$H values, but, as is well known, such hydroxides as those of aluminium and zinc become soluble in higher alkaline solution. This fact does not in any way contradict Fig. 4, but illustrates the point already mentioned in connection with the sulphides, namely, that the total metal in solution is often present in more than one form. Thus it is quite true that $[Al^{+++}]$

decreases with alkalinity ; at the same time $Al^{+++}$ ions can complex as follows

$$Al^{+++} + 3H_2O \rightleftharpoons AlO_3^{---} + 6H^+$$

In moderately alkaline solutions, the $[H^+]$ is still sufficiently high to avoid the formation of many aluminate ions, but the reaction progresses to the right as $[H^+]$ is reduced until, finally, most of the dissolved aluminium is in the form of aluminate ions and complete solution of the hydroxide occurs. However, many metals do not appreciably complex at high *p*H levels, and in these cases Fig. 4 gives an approximate indication of their hydroxide behaviour. All that has already been stated in connection with sulphide precipitation, i.e., complexing, (e.g., use of tartaric acid to prevent $Fe(OH)_3$ precipitation), effect of particle size on solubility, etc., is equally applicable to the hydroxides. It is interesting to note the effect of the addition of $NH_4Cl$ as well as $NH_4OH$ in precipitating hydroxides. At high *p*H levels most of the hydroxides are precipitated and little or no separation of the metals occurs (the separation of some of these will have already been made of course by virtue of the low solubility products of their sulphides, i.e., by precipitation in acid solution). The addition of ammonia alone, even in small amounts, will result in high *p*H values that are subject to marked change by evaporation, etc. On the other hand, when $NH_4Cl$ is employed, much more ammonia can be tolerated and at the same time the *p*H levels are reduced to values selective in hydroxide precipitation. Fig. 5 shows the results obtained by adding $NH_4Cl$ to aqueous ammonia solutions. Qualitatively the reactions are

$$NH_4OH \rightleftharpoons NH_4^+ + OH^-$$
$$NH_4Cl \rightleftharpoons NH_4^+ + Cl^-$$

For any concentration of ammonia, the addition of $NH_4Cl$ significantly increases the $[NH_4^+]$ and consequently reduces $[OH^-]$.

## ELECTROLYTIC SEPARATION TECHNIQUES

### Electrode Potential

When a metal is immersed in a solution of its ions, a potential exists between the metal and the solution

$$M_{\text{solid}} \rightleftharpoons M^+ + \varepsilon$$

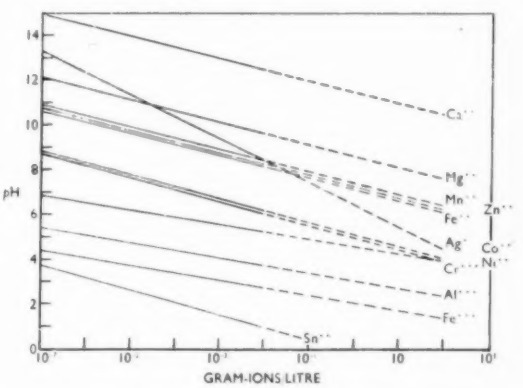


**Fig. 4.—Concentrations of ions in aqueous solution at various *p*H values in equilibrium with their respective hydroxides.**

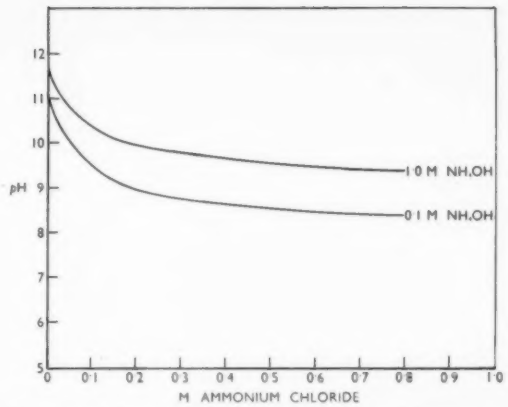


**Fig. 5.—Relationship between *p*H and concentration of ammonium chloride for various ammonium hydroxide levels.**

There is a tendency for metal cations to diffuse away from the electrode into the solution, and this continues until it is counteracted by the attractive electrical force between ions and electrode. At any temperature, a state of equilibrium then exists. The equilibrium potential is obviously dependent on the activity of the metal in the electrode (the activity is a maximum ($a = 1$) when a pure metal electrode is used, but can be lower when the electrode consists, say, of the metal dissolved in mercury), and also on the activity (i.e., concentration if the solution is very dilute) of the metal ions. Unfortunately it has not proved possible to measure the potential between an electrode and its solution without introducing unwanted potentials. Thus, to complete the electrical circuit for potential measurement, it is necessary to connect the solution to the voltmeter and the measurement includes not only the electrode potential required but also the potential existing between the solution and the probe. However, since practice is concerned mainly with the characteristics of complete cells (i.e., in which two or more electrodes are employed) differences in potential between one electrode and another are more important than absolute potential values between the metals and their ionic solutions. Consequently, it is customary to measure electrode potentials with reference to some standard electrode. A cell is produced, one half of which consists of the metal electrode and its solution of ions and the other half the standard reference electrode. The total voltage of the cell is measured and this is taken as the potential of the metal and its solution, since the absolute potential of the reference electrode is arbitrarily taken as zero. Several reference electrodes are in daily use, so that it is essential, when quoting an electrode potential, to define the reference, which may be a standard hydrogen or calomel electrode of various KCl strengths.

A typical cell for determining the electrode potential of zinc in a solution containing 1 gram-ion $Zn^{++}$ per litre (i.e., activity is approx. unity) would be

$$Zn/Zn^{++}\ (a = 1)\ //\ H^+\ (a = 1)\ /H_2\ 1 \text{ atmosphere}$$

At room temperature, the value is found to be 0·76 volts, and it is conventional to give such a voltage a positive sign if a positive current flows from left to right within the cell when the two electrodes are electrically connected external to the solution. Using this conven-

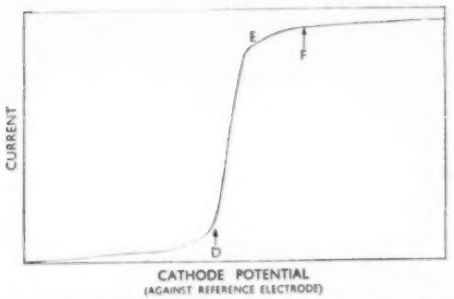


**Fig. 6.—Typical current/cathode-potential curve for metal deposition.**

tion, the voltage of the above cell is + 0·76 volts since the spontaneous reaction on joining the electrodes is

$$Zn + 2H^+ \rightarrow Zn^{++} + H_2$$

If, instead of short-circuiting the electrodes as above, a voltage opposing the + 0·76 volts is gradually applied, the above reaction will slow down and, assuming concentrations do not alter appreciably, no reaction will occur when the opposing voltage equals the + 0·76 volts in magnitude. Should the latter be exceeded by the applied opposition voltage, the reaction will occur from right to left, i.e., zinc is plated from solution and gaseous hydrogen dissolves to form $H^+$ ions. Thus a metal in contact with a solution containing ions of fixed activity creates a definite potential, and by applying a voltage greater or less than this potential the metal ions can be made to plate the electrode or the electrode to dissolve.

### Effect of Concentration on Electrode Potential

It is important to give some consideration to the variation of electrode potential with concentration of metal ions in solution. By thermodynamic arguments it can be shown that, at approximately room temperature, the E.M.F. of the above $Zn/H_2$ cell is given by

$$E_{S.H.E.} = E_o - 0{\cdot}0295 \log_{10} \frac{a_{Zn^{++}}}{a_{Zn}}$$

where $a_{Zn^{++}}$ and $a_{Zn}$ are the chemical activities of $Zn^{++}$ ions and the zinc in the electrode, respectively. For a pure zinc electrode, $a_{Zn} = 1$, and for reasonably dilute solutions, $a_{Zn^{++}}$ may be replaced by the concentration of zinc ions expressed as gram-ions/litre. $E_o$ is a constant and obviously equals $E_{S.H.E.}$ when $a_{Zn^{++}} = a_{Zn}$: this may be accomplished in practice by using a pure zinc electrode ($a_{Zn} = 1$) in contact with a solution containing 1 gram-ion/litre of $Zn^{++}$ ($a_{Zn^{++}} \backsimeq 1$). Thus for a pure zinc electrode

$$E_{S.H.E.} \backsimeq E_o - 0{\cdot}0295 \log_{10} [Zn^{++}] \qquad (8)$$

Equation (8) can be used to assess the effect on $E_{S.H.E.}$ of say a 100 times increase or decrease in concentration. $E_{S.H.E.}$ is calculated for $[Zn^{++}]$ and then $[100\ Zn^{++}]$, and by subtraction of the two $E_{S.H.E.}$ values it is found that a 100 times alteration in concentration results in a difference of $E_{S.H.E.}$ as low as approximately 0·06 volts. It appears, therefore, that, generally, a metal in contact with a solution of its ions containing say 1 gram-ion/litre produces a definite potential, and a small difference in this potential will produce either solution of the metal electrode or nearly complete removal of the metal ions by plating on to the electrode. The potential (with reference to the standard hydrogen electrode) at which the ions of a particular metal are removed from solution on to the pure metal electrode is approximately given by the $E_o$ value of (8). It is possible to arrange the metals in order of their $E_o$ values, and these should, in principle, give the order in which they are plated from a mixed solution on to a platinum cathode when the applied electrode potential is gradually varied.

### Current/Cathode-Potential Curves

So much for theoretical considerations; the results obtained in graded potential plating experiments must now be considered. If two platinum electrodes are dipped into a solution of copper sulphate, the potential of either may be measured with reference to a standard electrode (e.g., a calomel electrode) by having the latter in electrical contact with the copper sulphate solution—for example by means of a KCl bridge. The extremity of the bridge must be as near as possible to the electrode under examination. If now an increasing D.C. voltage is applied to the platinum electrodes, and at the same time measurements made of the current passing through the cell and the potential of the cathode (with reference to the standard electrode), the current-potential curve is of the type shown in Fig. 6. Little current flows until the point $D$ is reached (decomposition potential); at this point further increase in potential causes a marked current increase and copper plating definitely begins. With still further increases in cathode potential (obtained by increasing the total applied voltage to the cell) little gain in plating rate is obtained once point $E$ is passed. This is a consequence of diffusion control, and the limiting current is referred to as the diffusion current (polarographic analysis). The position of $E$ is a function of cathode area, and when this is small $E$ is reached at a low current value. Early measurements by Le Blanc (1893), of the potential corresponding to point $D$, using cadmium, silver, zinc and copper, confirmed that it coincided approximately with the static or thermodynamical equilibrium potential referred to above. It is actually slightly higher (i.e., more negative using the above-mentioned sign convention) due to polarisation at the cathode and the surface conditions of the cathode. This slight difference in potential, although present at the cathode during deposition of metals, has a much greater value when gases are evolved, and gives rise to the well known overvoltage effect. It is seen, therefore, that experimental work supports theory in that it is possible electrolytically to separate metals from solution by working at potentials slightly more negative than their decomposition potentials. In the case of Fig. 6, the optimum working potential would be close to $F$, since only small increases in separation rate would accompany more negative potentials, and there might be a danger of contaminating the electrode with the next metal to be separated. Electrolytic separations at fixed potentials for ionic separations have been employed for several years, and there are many designs of potentiostats in use with this technique. Some metals have decomposition potentials which are too close to allow easy straight electrolytic separation, and in some of these cases the difficulty is overcome by complexing one of the metal ions.

### The Mercury Cathode

It is interesting to consider the action of a mercury cathode in the light of the above. This is finding increasing use in metallurgical laboratories for the separation

of iron, etc., so that aluminium, titanium, vanadium and uranium may be chemically determined in the solution after electrolysis. Deposition of a metal at the mercury cathode is complicated by amalgamation; thus the reversible potential for $M^+$ ions is given by

$$E_{S.H.E.} = E_o - 0{\cdot}059 \log_{10} \frac{a_{M^+}}{a_M}$$

and whereas for an electrode of pure M, $a_M = 1$, an amalgam of M will usually have an activity $a$ less than unity, its precise value depending on the amount of metal deposited. This effect is illustrated by sodium and potassium, whose deposition potentials at a mercury cathode are nearly 1 volt smaller than for the solid metal electrodes. Because of the amalgamation effect, it is not possible to obtain the correct deposition potential for a metal at a mercury cathode without practical measurement. Lingane, Swain and Fields have shown that ordinary polarograms of the solution to be electrolysed give excellent values of the optimum control potentials for a large working mercury cathode. The half wave polarographic potentials of the various metal ions are first measured (with reference to a standard electrode) and the control potentials are then taken as being about 0·1 volt more negative in each case.

Consideration of the mercury cathode must not be left without reference to overvoltage. It is fortunate that the overvoltage for hydrogen is approximately 0·8 volts at a mercury surface, since this results in more metals being deposited before the vigorous evolution of hydrogen sets in. The following half wave potentials are taken from polarograms of neutral or slightly acid solutions:

Approximate Half Wave Potentials (S.C.E.)

| | | | |
|---|---|---|---|
| Hg | >0·4 volts | $Mn^{++}$ | −1·51 volts |
| $Fe^{+++}$ | ∞0·2 ,, | $Cr^{+++}$ | −1·53 ,, |
| $Cu^{++}$ | ∞0·0 ,, | $H^+$ | −1·58 ,, |
| Mo | −0·34 ,, | | (includes approx. −0·8 volts overvoltage) |
| Sn | −0·5 ,, | | |
| Ni | −1·1 ,, | Zr | −1·65 volts |
| $Co^{++}$ | −1·25 ,, | Al | −1·75 ,, |
| $Fe^{++}$ | −1·3 ,, | | |

By passing a current through an electrolyte containing the above metals, those with more positive half wave potentials than hydrogen will enter the mercury. Those below hydrogen are in effect protected by hydrogen formation, and include aluminium and zirconium, which are known from experiment to remain in solution. Without overvoltage, hydrogen evolution would become excessive at approximately − 0·8 volts and this would seriously interfere with the deposition of the metals from nickel downwards.


### Acknowledgment

The author would like to express his appreciation to Mr. R. A. Hacking, O.B.E., M.Sc., F.I.M., Director of Research of the R.T.S.C. Laboratories for encouragement and facilities during the preparation of this paper.


# A Solution Method of Spectrographic Analysis


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*County Technical College, Wednesbury*

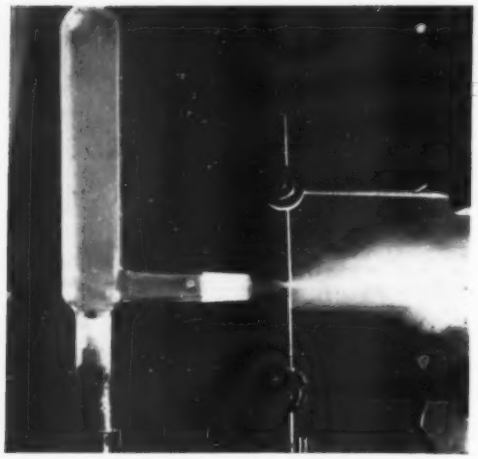

QUANTITATIVE methods of spectrographic analysis depend upon the determination of the relative intensities of selected spectral lines, emitted by a light source containing the sample to be analysed. The choice of light source is governed by several factors, such as the quantity and nature of the material available and, in general, quantitative methods of spectrographic analysis currently applied involve the excitation of the solid sample by arcing or sparking against a counter electrode.

It is significant that whilst the spectrographic analysis of low alloy steels is satisfactory, the same accuracy cannot be claimed in the case of the light alloys. This would seem to indicate that errors introduced by the sample material, due to segregation and compound formation in the alloy, are more important than those arising from the manner of excitation and the measurement of spectra.

One method of eliminating errors arising from sample inhomogeneity would be to employ a solution method of analysis. Other advantages accruing to a solution method would be that samples in the form of turnings or drillings could be readily handled, the necessity of casting and preparing pencil electrodes would be eliminated, and, finally, accurate standards could be synthesised from analytical quality reagents or from solutions of spectrographically pure metals.

Since it is well-known that spark methods of excitation are much more reproducible than arc methods, the known techniques for sparking solutions were examined. Residue methods are limited in scope since most types of deposit are thrown from the electrode immediately upon striking the spark, and impregnation techniques are not generally satisfactory due to the wide variation in the extent to which the same solution is absorbed by different graphite electrodes. Capillary flow methods do not adequately charge the electrode surface, and "forced flow" methods require relatively large quantities of liquid, sputtering is excessive, and, of greater importance, the reproducibility of the intensity ratios of selected line pairs is less than that obtained from solid samples.

The porous cup technique described by Feldman[1] was next examined. The electrodes were charged with the solution by means of a hypodermic syringe so that the electrode cavity was filled from the bottom and was consequently free from trapped air bubbles. Nevertheless, during sparking expansion of air from the side walls of the cavity caused the solution to spill over the top of the cup electrode. Pre-soaking the electrodes effected no improvement, and the difficulty was finally overcome by inserting a small diameter refractory rod into the cup: during sparking liquid displaced by air expansion was carried by surface tension effects up the refractory



**The Lundegardh atomiser in operation.**

rod, eventually draining back into the graphite cup. Though the difficulty of spillage of solution was overcome in this way, the reproducibility of intensity ratios of selected spectral line pairs was not considered to be of a sufficiently high order to form the basis of an improved analytical technique for the general run of metallurgically important alloys.

It had previously been found[2] that quite satisfactory spark spectra of refractory materials ($SiO_2$, $Al_2O_3$, etc.), could be excited when the powdered material was allowed to fall freely through hollow graphite electrodes forming a spark gap. Working on these lines, an alloy solution delivered in the form of a fine spray through a hollow graphite electrode forming the counter electrode of a graphite/graphite spark-gap was found to give well resolved spectral lines. However, it was found that after a period of sparking, incrustation of the electrodes became so severe that the channel was completely sealed. Spraying the liquid directly into the spark-gap was tried and, again, well resolved spectral lines were obtained which upon evaluation were promisingly consistent: this agrees with the findings of Lamb[3] who used this technique for the analysis of biological fluids.

In order to evaluate the scope and accuracy of the method, it was decided to apply it to the same alloy system and wide concentration range for which data obtained by the Feldman technique existed.[4] Accordingly, a number of copper-zinc solutions were made up from spectrographically pure copper and zinc by dissolving 1 g. "alloy" in 10 ml. acid (50% $HNO_3$) and diluting to 100 ml. These solutions were then sprayed into the spark gap, each solution being exposed in duplicate on each of two plates, which were then photometered. The results were so promising, and in view of the fact that they were obtained with a relatively coarse spray from a manually operated atomiser, it was decided to continue the work using a Lundegårdh atomiser.

The Lundegårdh atomiser was operated through a reducing valve from a nitrogen cylinder at 30 lb./sq. in. pressure, the rate of consumption of solution being 6 ml./hr. Under these conditions an exceptionally fine spray was delivered from the side arm (Fig. 1) which did not lose its cylindrical form over several centimetres from the orifice. Since the diameter of the spray delivery tube was 12 mm. and the analysis gap was 4 mm., it was

TABLE I.

| Sample | % Zinc | |
|---|---|---|
| | Chemical (by difference) | Spectrographic |
| 1 | 29·00 | 28·80 |
| 2 | 24·60 | 24·30 |
| 3 | 28·20 | 28·00 |
| 4 | 21·16 | 22·00 |
| 5 | 29·27 | 29·85 |
| 6 | 33·19 | 33·45 |
| 7 | 22·50 | 22·33 |
| 8 | 20·45 | 21·17 |
| 9 | 22·50 | 22·42 |
| 10 | 20·95 | 21·55 |
| 11 | 29·20 | 28·60 |

easily arranged that the spray column should fill the spark-gap. The distance of the spray delivery tube from the spark-gap was found not to be critical over a distance of 2 cm. but was arranged constant at 1 cm.

Reproducibility tests were carried out using different types of photographic plate, different sizes of electrodes and electrode materials, conditions being finally standardised as follows:—

| | |
|---|---|
| Slit-Source Distance | 30 cm. |
| Spark-Gap (4 mm diam. graphite electrodes) | 4 mm. |
| Source Unit | 0·35 kVA uncontrolled condensed spark: no added inductance |
| Photographic Plate | Kodak B20 |
| Slit Width | 0·015 mm. |
| Slit Length | 2 mm. |
| Exposure Time | 40 sec. |
| Line Pairs | Zn 2502/Cu 2544<br>Zn 2557/Cu 2544 |

For calibration purposes the solutions containing up to 50% Zn were exposed in triplicate on a single plate, the mean log-ratios being used to construct two calibration curves based on the line pairs indicated above.

A brass sample containing 27·66% Zn was exposed 133 times (nineteen times on each of seven plates), plate calibration being effected by three exposures on each plate of a 70/30 solution. The results obtained were as follows:—

| | |
|---|---|
| Standard Deviation | 0·38 (in terms of Zn content 1·37%) |
| Highest Figure | 28·85% Zn. |
| Lowest Figure | 26·85% Zn. |

Taking the mean of three determinations the results were:—

| | |
|---|---|
| Standard Deviation | 0·23 (in terms of Zn content 0·83%) |
| Highest Figure | 28·30% Zn |
| Lowest Figure | 27·16% Zn |

As a final check on the method various samples of commercial brasses were examined spectrographically and the results compared with independent chemical analyses. The results are shown in Table I.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## Correction

"PHOTOMETRIC DETERMINATION OF ZIRCONIUM IN HIDUMINIUM RR.350 AND SIMILAR ALLOYS."

We regret that an error appeared in the above article which was published in our March issue. The first line of the second column on page 158 should read:—

"hydrochloric acid from a burette, add 10 ml. of dye solution and dilute to 25 ml."

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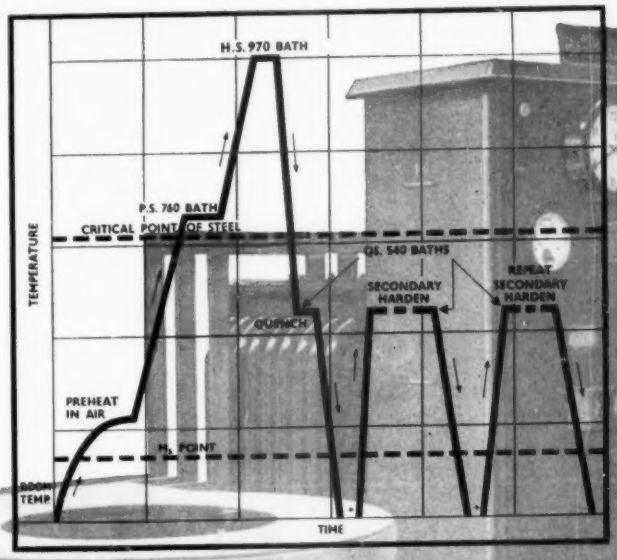


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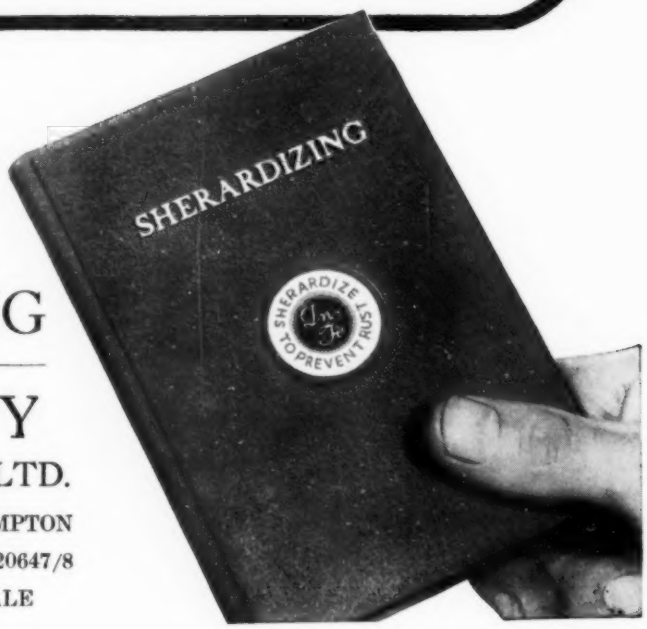


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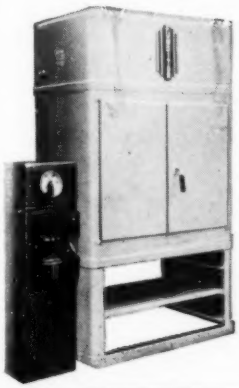

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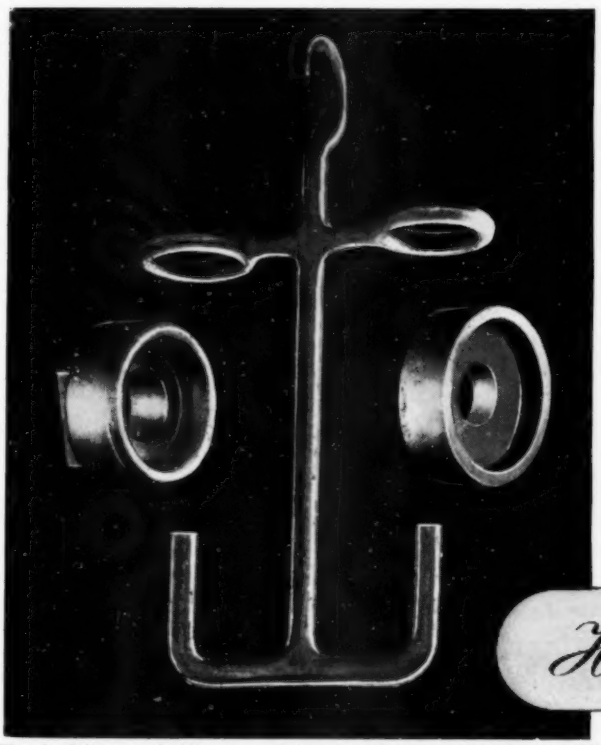

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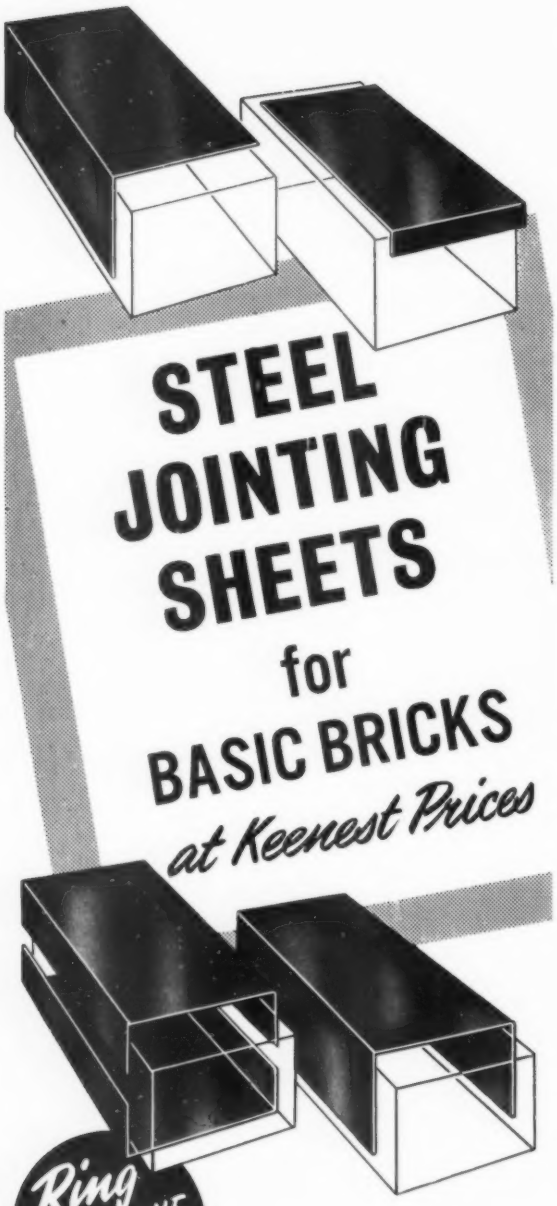

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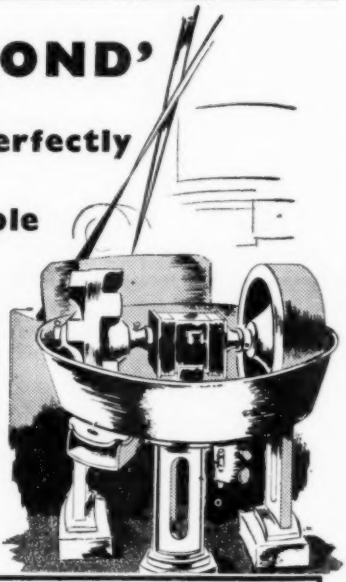

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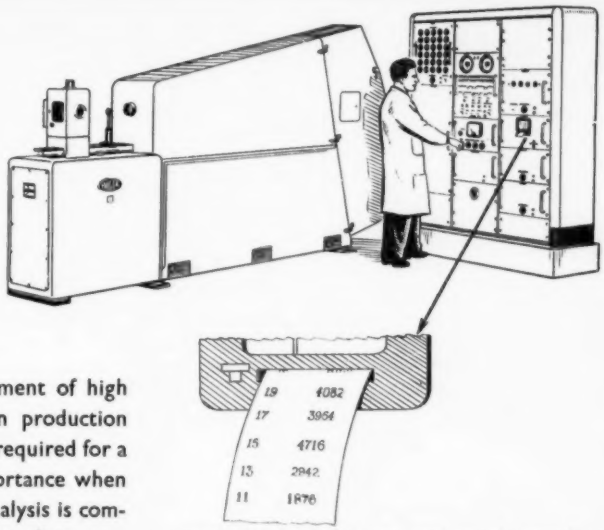


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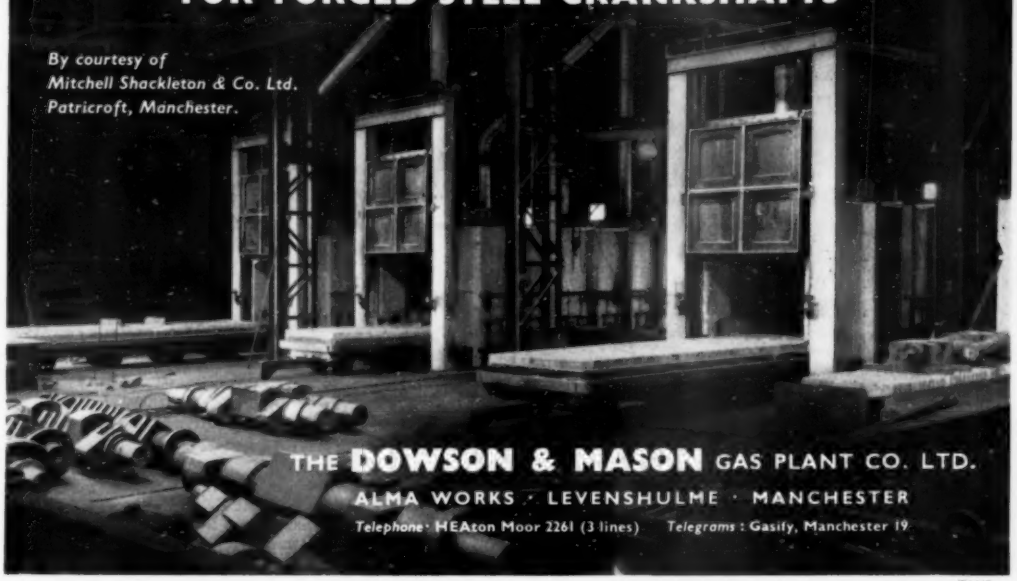

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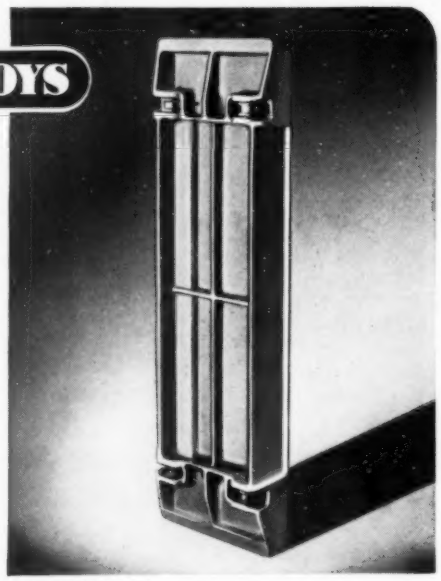

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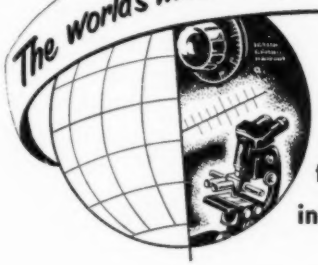

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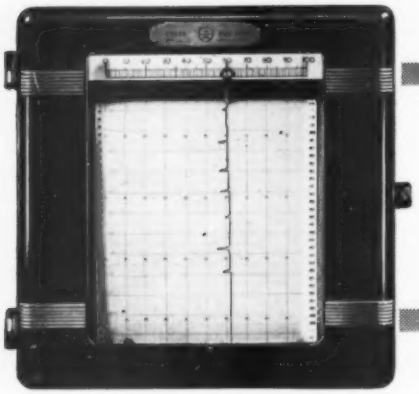

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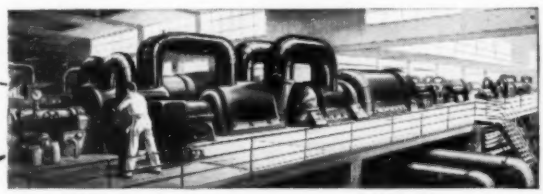

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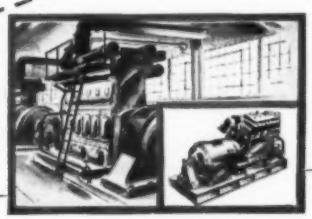

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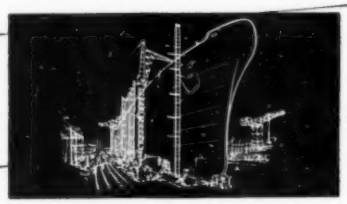

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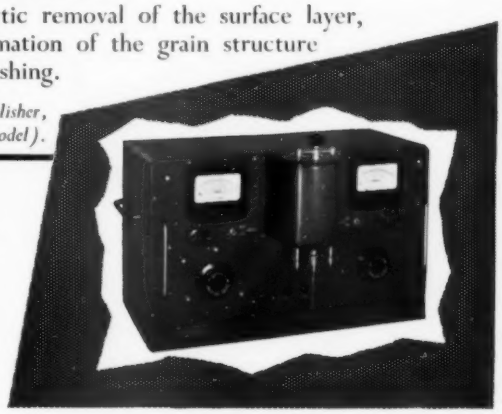

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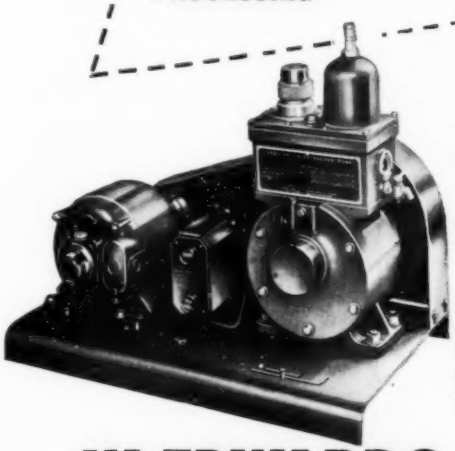

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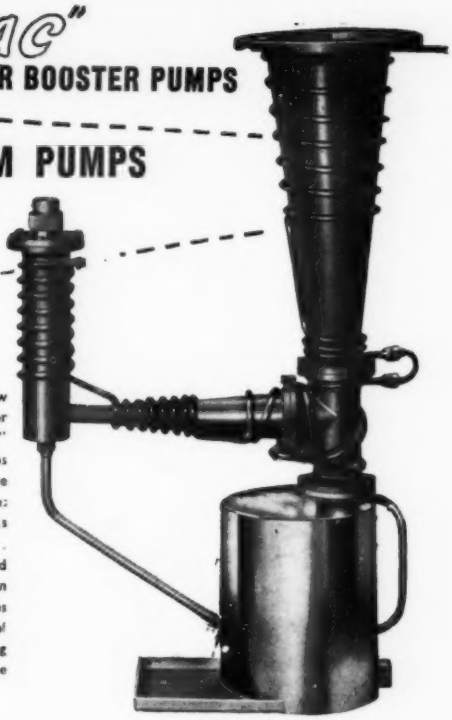

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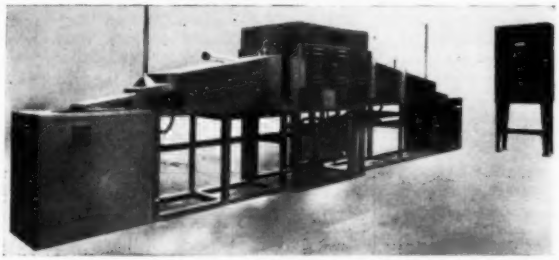

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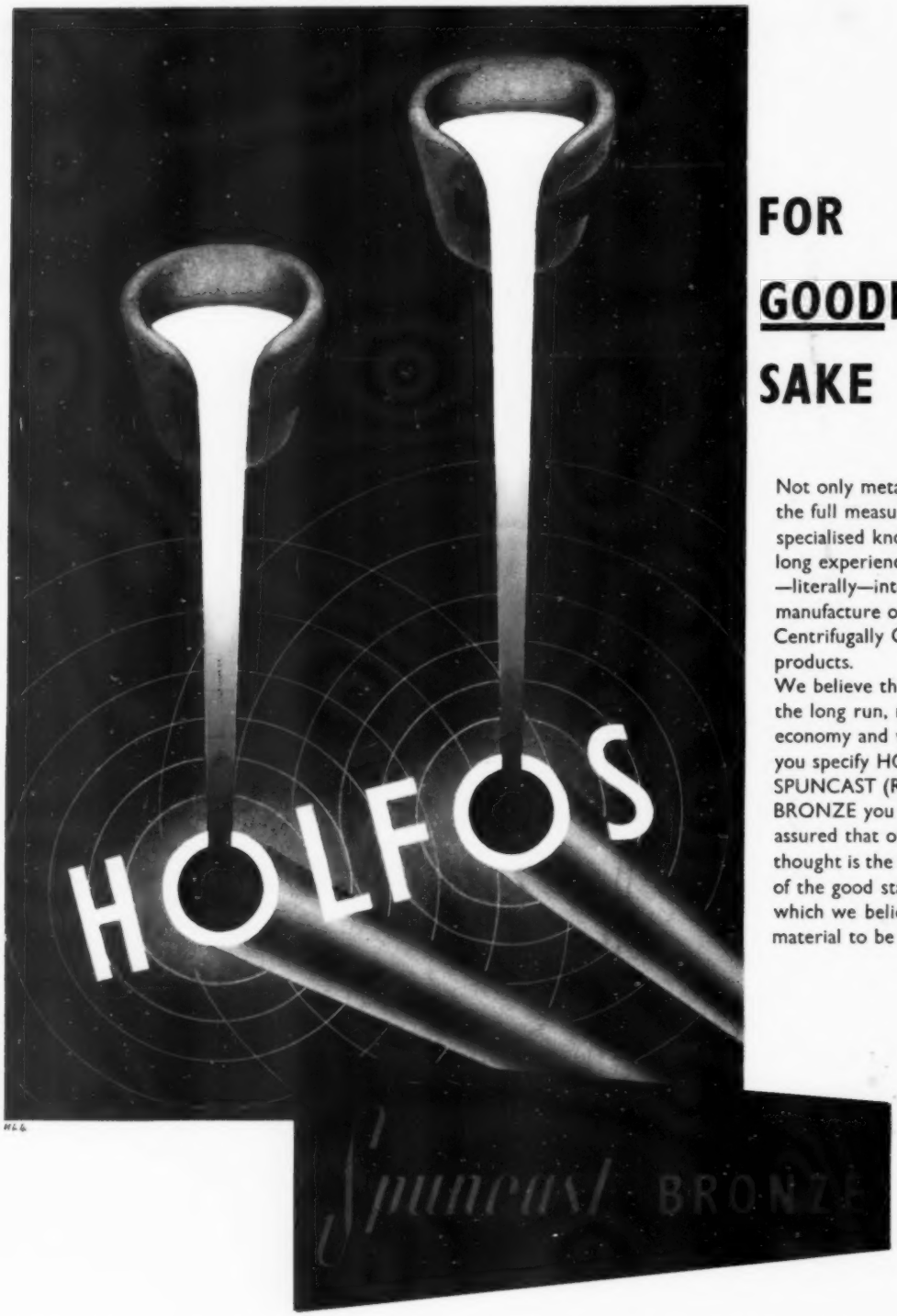


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